

Twenty-Year Retrospective of National Center for Preservation Technology and Training Sponsored Archeology



National Park Service
U.S. Department of Interior

National Center for Preservation Technology and Training



# Proceedings of the Twenty-Year Retrospective of National Center for Preservation Technology and Training Sponsored Archeology



Edited by: Tad Britt, NCPTT

Proceedings of the Twenty-Year Retrospective of the National Center for Preservation Technology and Training Sponsored Archeology Symposium
San Francisco, California, April 18, 2015
Edited by Tad Britt, NCPTT

Organized by

The National Center for Preservation Technology and Training (NCPTT)

In Association with

The Society of American Archaeology

The National Center for Preservation Technology and Training (NCPTT)

645 University Parkway

Natchitoches, LA 71457

May 19, 2017

ISBN:

Electronic PDF Format 978-0-9970440-3-4

Since 1994, the National Center for Preservation Technology and Training, a program of the National Park Service (ncptt.nps.gov), has engaged in state-of-the-art research in archeological treatments and technologies. The Center provides grants, education, research, and training opportunities in the areas of archeology and collections, architecture and engineering, materials conservation, and historic landscapes. To date, over \$10 million dollars have been spent on sponsored research via our grants program. This symposium is a 20 year retrospective and is focused on the innovative contributions of the award recipients to the archaeological sciences, methods, and technologies.

Specifically, the authors were asked to re-examine their original work and address the impact of their research on their respective fields; how their work has influenced their research; and progress in their study areas since their initial award. These proceedings include topics ranging from the development and fielding of magnetic susceptibility, archaeogeophysics, and a friction cone-penetrometer, to plasma extraction 14C analysis, site location probability models, ceramic thin-section analysis, freshwater shell artifact and temper sourcing, and Native American consultation protocols.

The papers and presentations included here reflect the depth and breadth of the types of studies funded by NCPTT. The materials herein are presented in their entirety in their original format with minor editing. We hope you find the proceedings interesting and informative, and look forward to the next twenty years. Special thanks to Tad Britt, Chief of NCPTT's Archeology & Collections program, who conceived and organized this retrospective. Thanks also to both the authors and readers for your contribution and patience in getting the publication ready.

Kirk A. Cordell Executive Director National Center for Preservation Technology and Training April 2015

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#### Native Americans and Archaeology Training Workshop: A Twenty-Year Retrospective

Kurt E. Dongoske, RPA Zuni Cultural Resource Enteprise

The Arizona Archaeological Council received funding from the NCPTT during its inaugural granting cycle to conduct a two-day training workshop between Native Americans and archaeologists. The goal of the workshop was to promote a productive dialogue between Native Americans, Federal agency archaeologists, academic archaeologists, and archaeologists from the contracting community. Three issues were the focus of that workshop: consultation, oral tradition and archaeological interpretation, and Native Americans' role in archaeology. This presentation reviews the proceedings and the products of that workshop followed by an evaluation of the current condition of the relationship between Native Americans and archaeologists and what progress, if any, has been made in the twenty years since that workshop.

#### Introduction

In 1994, the National Park Service's National Center for Preservation Technology and Training (NCPTT) funded the Arizona Archaeological Council's grant proposal, entitled "Native Americans and Archaeology Training Workshop." The grant was one of fifteen grants awarded in the NCPTT's first granting cycle.

The primary objective was to conduct a two-day training workshop designed to promote communication and understanding between archaeologists and the Native American community. The workshop was structured as a two-day dialogue between Native American tribal representatives, federal agency archaeologists, academic archaeologists, and archaeologists from the contracting community to address three topics: 1) consultation, 2) oral tradition and archaeological interpretation, and 3) Native Americans' role in archaeology.

This presentation provides an account of the proceedings and results of the 1994 workshop followed by personal observations concerning the evolving relationship between Native Americans and archaeologists that has occurred over the past twenty years since the workshop was held.

#### The Workshop

The Native Americans and Archaeology Training Workshop was held on 9 & 10 November 1994 at the Woodlands Plaza Hotel in Flagstaff, Arizona. The Bureau of Reclamation, Upper Colorado Regional Office, graciously provided a professional facilitator for the workshop. The participants in the workshop consisted of Native American representatives from the Hualapai Tribe, Hopi Tribe, Navajo Nation, and Pueblo of Zuni. Federal and state agency archaeologists represented the Department of the Interior, Office of the Secretary; the Coconino National Forest, Office of Navajo and Hopi Indian Relocation; Arizona State Historic Preservation Office; and Arizona Department of Transportation. One archaeologist represented a public utility provider. Archaeologists affiliated with academia were from University of Arizona, Arizona State University, and Northern Arizona University. Archaeologists from the contracting community were represented by Desert Archaeology, Institute for the North American West, and Soil Systems, Inc.

Each participant in the workshop was invited because they possessed a significant amount of experience and knowledge on the identified discussion topics. The workshop was designed to provide a venue for archaeologists and Native Americans to share ideas and concepts in order to foster an understanding of each other's perspective, limitations, and expectations. In addition, the workshop anticipated that a mutual understanding between federal agency archaeologists and Native American representatives could be

#### Native Americans and Archaeology Training Workshop: A Twenty-Year Retrospective

achieved regarding consultation, oral tradition and archaeology, and Native Americans' role in archaeology.

The following is a brief synopsis of the discussion on each of the identified topics that took place during the workshop.

#### Consultation

The discussion among the workshop participants regarding the issue of consultation was complex and represents the greatest amount of time spent on any one of the three topics. In general, the workshop participants agreed that the history of consultation between Tribes and federal agencies represented a broad range that included a sincere and earnest attempt to solicit the concerns of the tribe to the more customary, impersonal, cursory federal form letter stating "... if we don't hear from you in 30 days we will assume concurrence." Consultation requests were separated into two categories, one category consultation with the capital C represents the legally required consultation that is framed by federal legislation, executive orders, and Presidential memoranda; whereas, the other consultation with a lower case c represented consultation inspired by professional archaeological codes of ethics.

Consultation required by federal law can be mutually rewarding when it is fashioned by positive personal working relationships developed over time between the federal agency representative and a tribal representative. It is these personal relationships that are really at the heart of effective consultation. It was acknowledged, however, that personnel, both within an agency and a Tribe, can change based on career advancement, career moves, and tribal elections, and that these changes can have a detrimental effect on subsequent consultation efforts.

Concern was expressed about the ever increasing number of requests from federal agencies, and to a lesser extent state agencies, for consultation and that this increase places an enormous burden on a Tribe; tribal representatives labeled it as a "paperwork blizzard." A suggested method for dealing with these overwhelming and increasing requests for consultation was to have each Tribe

define the geographic extent within which they would like to be consulted. It was emphasized that the geographical area in which a Tribe wants to be consulted should be determined by that Tribe and not by a federal agency individual utilizing land claims commission boundaries.

Effective and meaningful consultation must acknowledge, respect, and sensitively negotiate the world views held by Tribes which diverge from those of the Western scientific ontology that is embedded in federal agencies. Often, conflicts of cultural value stem from these differing ontological perspectives and how cultural resources are defined which lead to a breakdown in communication and a decrease in the resultant quality of the consultation. Federal agencies should also recognize that Tribes can and will classify cultural resources as those resources that federal agencies routinely classify as natural resources.

The workshop participants offered suggestions for improving the consultation experience. In order to streamline the consultation process federal agencies should consider working with Tribes to develop agreement documents for routine and repetitive federal actions. These agreement documents could specify coordinating and collaborating mechanisms between the agency and the Tribe, as well as identify individuals directly responsible for the consultation process. In addition, regularly planned sensitivity training for federal and tribal representatives could be a stipulated annual occurrence identified in the agreement document.

Transparency in the process is vital to successful consultation, including providing the Tribe with a clear understanding of how their expressed concerns were considered and acted on as part of the federal decision-making process, and that the project plans were revised, if necessary.

Finally, ensuring confidentiality of tribal information considered esoteric and culturally sensitive should be a primary concern of the federal agency. The workshop participants felt it was necessary for federal agencies to develop policy statements that define what types of information are sensitive and restricted from public access. There was

a perceived need for legal protection of ethnographic data that would be comparable to archaeological site location information. The workshop participants also felt that it was naïve of federal agencies to think that a tribal representative should possess all the knowledge about sacred sites and/or traditional cultural properties and that ethnographic research should not be required to produce this type of information. This type of view of the Tribes places a greater level of accountability on the Tribe than is currently expected of federal land managers.

#### Oral Tradition and Archaeology

Archaeologists are interested in learning about the past. Native Americans are interested in maintaining the cultural traditions they inherited from their ancestors who lived in the past and in many instances created the archaeological record. For Native American tribes with strong oral traditions, their primary sense of history comes from the narratives, stories, and accounts passed down by tribal elders. The workshop participants discussed whether archaeology and Native American oral tradition can benefit each other and if they actually came together at some point. For example, archaeologists work to reconstruct the past and are interested in how oral tradition can contribute to the reconstruction of the past, whereas, for many Native Americans, oral tradition is good for explaining and directing life histories and it directly informs the individual about the proper way of conducting one's self.

Workshop participants agreed that utilizing oral tradition to inform archaeological research was a delicate ethnographic issue. Tribal representatives expressed a concern that archaeologists have mined the archaeological record over the past 100+ years and now are interested in mining oral tradition. The tribal representatives were also cautious on how oral tradition would be employed in archaeological research because by and large, they perceived archaeologists as scared of Indians, which contrasts with physical anthropologists who are petrified of Indians, and social anthropologists who are not even thinking about working with Indians.

In response to these expressed concerns, the workshop participants felt that Tribes should set their own research agendas with the knowledge that these research agendas will vary by Tribe. In addition, each Tribe should develop its own set of protocols to guide and control outside researchers' access to tribal members and the manner in which ethnographic information may be utilized and disseminated by the researcher.

One tribal representative stated that Native American female informants were a too-often neglected source of insight into the past. For example, if an archaeologist wanted to do research on archaeologically defined rooms within a room block or pueblo, then this tribal representative suggested they really needed to talk to females, not males. The workshop group agreed that, to successfully incorporate oral tradition into archaeological research, it will be necessary to develop elaborate archaeological research ethics. As a part of those ethics, Native American participants who provide ethnographic information to archaeologists should be provided the opportunity to co-author research reports and journal articles. Archaeologists who utilize tribal ethnographic information in their research should be ethically obligated to reciprocate something back to the Tribe. Examples of this reciprocity were working to educate tribal members about the research, to guarantee too that tribal and individual cultural advisor's rights are observed, and to ensure materials (video, audio tapes, and research notes) generated during ethnographic research are appropriately archived to tribal requirements. As an incentive toward developing a ethical research statement, the following five recommendations were generated regarding the use of oral tradition in archaeological research:

- 1. Determine by asking tribal officials whether or not a Tribe wants its oral traditions used in archaeological research.
- 2. If tribes want oral traditions to be used in archaeological research, then archaeologists need to establish the parameters of that use with Native

#### Native Americans and Archaeology Training Workshop: A Twenty-Year Retrospective

American cultural advisors and tribal officials. This needs to be done at the outset of the research.

- 3. Tribal cultural advisors are subject specialists who should be compensated for their time (like other professional researchers) on funded cultural resource projects.
- 4. If tribes do not want oral traditions used in archaeological research, then archaeologists should state this in reports. These reports should acknowledge that the review of culture history and the scientific findings do not include oral traditions at the request of the Tribe.
- 5. Encourage tribal review of archaeological research, especially if it uses oral traditions.

#### Native Americans' role in archaeology

The final topic the workshop participants were asked to consider was Native Americans' role in archaeology. That is, do Native Americans really want a professional role in archaeology? If the answer is no, then what utility do Native American groups find in the discipline of archaeology and its associated research methods? One tribal representative pointed out that the Zuni Tribe has one of the longest lived archaeology programs operating in the United States. The Zuni Archaeology Program brings many tribal members into the profession of archaeology because it fits with individual Zuni's family need for a job on the Zuni Indian Reservation and through it an individual Zuni can gain marketable skills. However, currently Native Americans working in archaeology as laborers or skilled crew members has an unintended consequence of creating a glass ceiling for themselves because most Native Americans do not have a university degree making it difficult, if not impossible, to get the needed skills and advance to a high position.

While there was a perceived need, by tribal representatives and archaeologists, for archaeologically trained Native Americans on Indian reservations, the question that remained was how to break this glass ceiling by successfully transforming practical archaeological field work experience into college course credit. For many Native American youth, especially those with families to support, it is hard, if not impossible, to drop everything and go off the reservation to a university for four years to get a college degree. The university systems needed to be more sensitive and reactive to the educational needs of Native Americans that live and want to stay on the reservation by providing off campus learning opportunities and college credit.

#### **Workshop Products**

The workshop produced several products: (1) a white paper was developed on Native American oral tradition and archaeology written by four of the workshop participants. This white paper was first published in the SAA Bulletin in March of 1996 and later republished in 2000 in an SAA publication Working Together: Native Americans & Archaeologists. A revised version of this paper was published in the 1997 publication Native American and Archaeologists: Stepping Stones to Common Ground published by Altamira Press; (2) training workshop proceedings' notes, along with flip chart notes, were word processed and provided to the NCPTT; (3) seven audio tapes of the workshop proceedings were generated of which three of the tapes have been transcribed.

#### **Summary and Conclusions**

In the twenty years since the Native Americans and Archaeology Training Workshop was held there have been many changes and developments, mostly positive, in the relations between Native Americans and archaeology. Many of these changes are self-evident within the Society for American Archaeology, such as, the SAA's Native American Scholarship Fund, the standing Committee on Native

American Relations, and the increasing number of Native Americans that are becoming members of the SAA.

There has also been a concurrent issue in the number of Native American women earning Ph.D.s, entering academia, and positively impacting how archaeology is taught to a new generation of emerging archaeologists. This has resulted in increasing the feminine voice in archaeological research.

The last twenty years has also witnessed an increase in interest in the successful integration of Native American oral tradition into archaeological research, especially in explaining prehistoric migration in the Southwest.

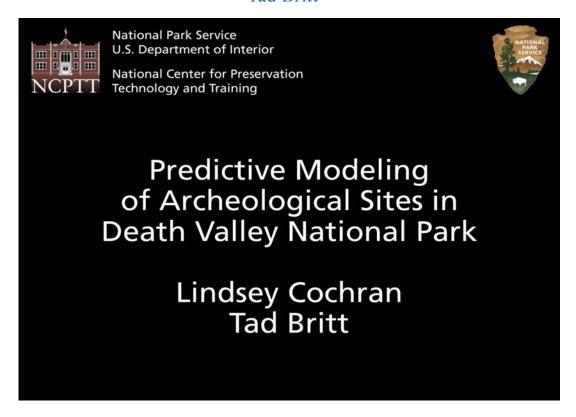
Consultation, on the other hand, may not have undergone as bright a transformation as the other topics. As noted by the workshop participants twenty years ago, successful consultation works only when the individuals who carry out that consultation are personally invested and committed to its success through mutually appreciating and respecting the different cultural perspectives and values that are involved. This situation continues to be even truer today. Unfortunately, it has been my experience that this situation remains the exception and not the rule. Tribes continue to receive numerous requests for consultation that contain the same repetitive bureaucratic form language stating the federal agency assumes tribal concurrence if the Tribe does not respond in thirty days. In spite of the fact that "Tribal consultation," though explicitly insisted upon by the President in orders to the agencies and in his approval of the 2010 United Nations Declaration on the Rights of Indigenous Peoples, many federal agencies continue to treat tribal consultation as a "check the box" approach to compliance. One can only speculate that perhaps there is too much inherent bureaucratic inertia within these federal

agencies that resist any efforts to change the business as usual approach to consultation.

This unsatisfying climate of tribal consultation continues to be a recognized issue that is in serious need of attention and revising. The importance of this issue is underscored by the fact that the Arizona SHPO, Tribes, and federal agencies are currently planning a tribal consultation workshop that precedes the Arizona state historic preservation conference next month. Unfortunately, federal/tribal consultation as it is currently structured continues to have the unintended consequence of imposing colonialism on Native American Tribes because the decision-making process is not mutually achieved between two negotiating sovereign powers, but rather it remains a unilateral decision made by a settler government.

Although there is still much to do to improve the relationship between Native Americans and archaeologists, the Native Americans and Archaeology Training Workshop represents a snapshot in time and is a useful contrast to demonstrate how much we have achieved in improving the relationship between archaeologists and Native Americans. In closing, I would like to extend my sincere appreciation to the folks at the National Center for Preservation Technology and Training for having the sensitivity and vision in seeing the benefits of funding the Native Americans and Archaeology Training Workshop.

Lindsey Cochran
Tad Britt



This study employs spatial modeling and earth and social sciences to integrate environmental variables relevant to past and changing landscapes. The purpose is to determine probability models for different types of archeological site types and their locations at probable locations distributed across the landscape at DEVA.

Objective: To develop a set of archeological site type specific models of a location probability statistics AND develop a GIS enabled database for day-to-day analysis, as well as long-term management operation strategies.

The purpose of this modeling effort is to:

- Develop and compile a GIS database specifically for the modeling process
- Analyze spatial data using statistical tools to

- assess the relationship between environmental factors and the location of known archeological sites
- Improve understanding of the relationships among environmental variables and archeological locations
- Create scalable (space, time, and behavioral)
  models of potential past land use through taking
  into account the spatial relationships and key
  characteristics of known locations
- Make recommendations to managers to better focus areas for surveys and the monitoring of sensitive archeological site locations
- Test and improve the models through an iterative process of comparison with future survey results and evaluation and refinement based on those results

# Objectives: Ecological niche models of archeological sites

- Develop the geographical landscape data for the models
- Organize the archeology into site types for model development
- Estimate probability of sites being located in various places in the Park using MAXENT, an ecological niche modeling system
- Interpret and provide an overview of results from the probability models

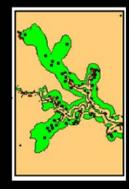
To accomplish these goals, from collated data on known sites, the authors assembled a set of Geographic Information System (GIS) map layers and developed models using a niche modeling approach with the software Maxent (Elith et al. 2011, Peterson 2006, Phillips S. 2006. Ecol. Model. 190: 231). This ecosystem approach requires grid-based GIS maps depicting important

landscape characteristics and the location of known archeological sites. The sites included in the models described here were generated from the records database supplied by the NPS archeological office. The landscape factors included in these models were selected to define the terrain and access to water using spatial data that were already available.

#### **General Approach**



IF human behavior is random, THEN the Arch. sites might be scattered across the landscape without a pattern



BUT we know that human behavior is patterned, THUS the natural and social environments influence the chance that a site will be in particular types of places

We can use statistical measures that find the patterns of archeological sites relative to a set of landscape features stored as GIS layers.

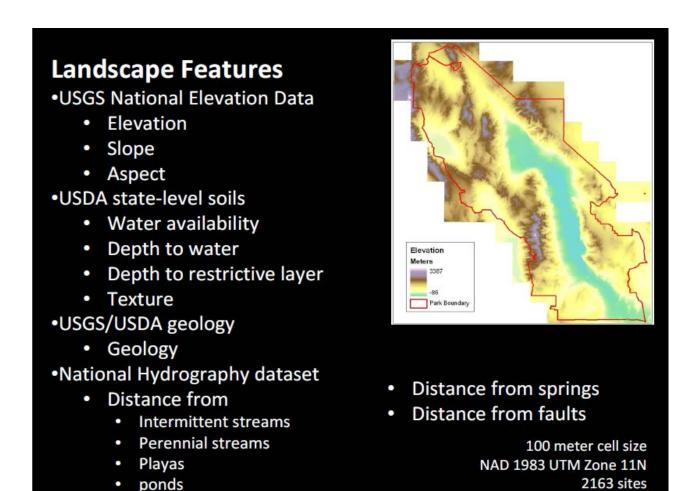
#### From this:

- 1. We can determine where on the landscape, things were likely to have occurred based on existing sites so that we can better approach surveys for new sites
- 2. We learn more about how people were interacting with the environment and find additional clues about possible changes in physical features over time

Methods: "A predictive model--that is, an array of correlations, and their strengths, among locations and environmental variables in a study area" (Ebert 2000).

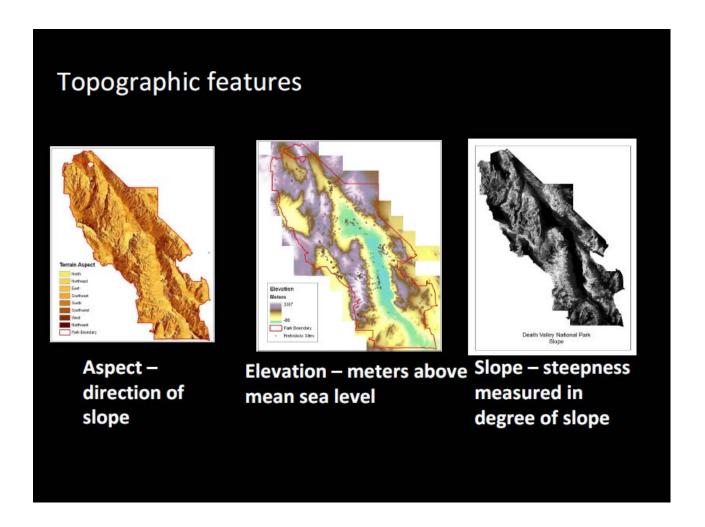
We began, with information from DEVA, our initial assessments and data processing for the development of the spatial models of archeological site potential based on past human use. This effort includes the use of an ecological niche model of past landscape use with the modeling program

Maxent. The major activities will be to: (1) Develop a structured database of archival information of the existing site records that populates the requirements of the landscape model; (2) Process existing geographic data from currently available digital archives to be used in the ecological niche model; and, (3) Analyze and interpret the output from the niche model to assess associations between existing landscapes and sites and to prepare more detailed requirements for the data development needed to provide data for a more directed modeling effort.



The cultural understanding of sites was refined by application of geomorphic principles. Characterization of landform surface dynamics and deposit age allowed us to integrate geologic and landscape elements into the site distribution models.

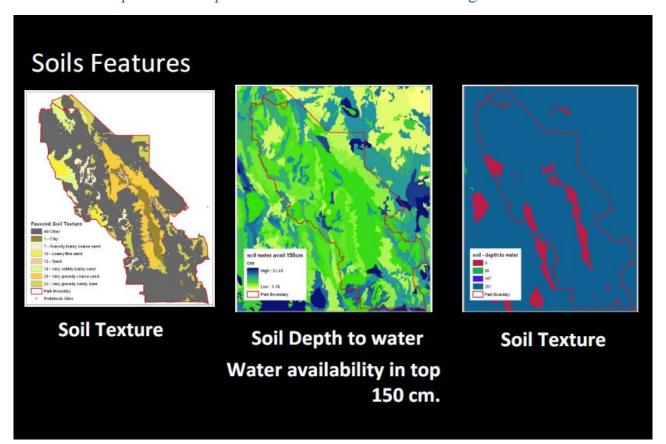
The geomorphology associated with the landscape factors provided key insights into the interpretation of the terrain, soils, geologic and hydrologic features associated statistically with the site distributions.



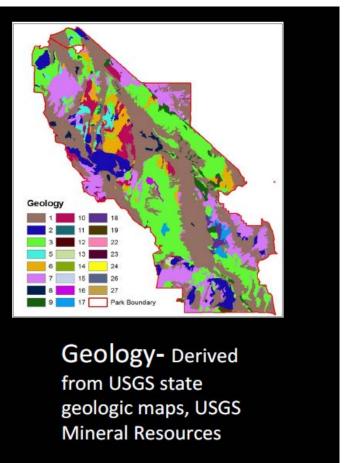
#### Landscape Features

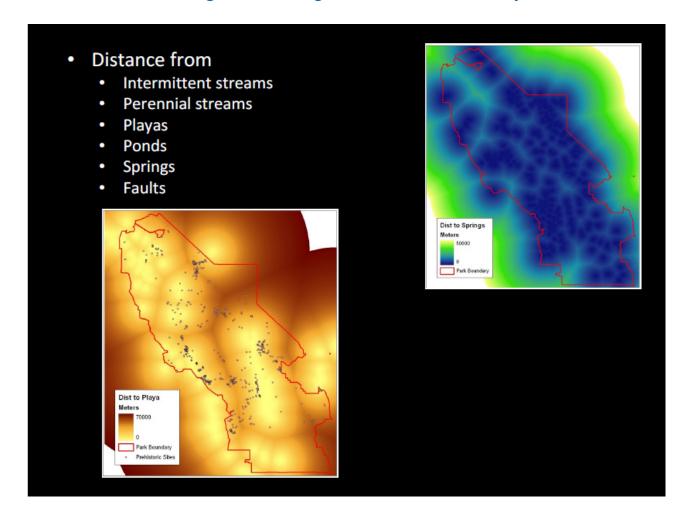
A set of landscape features was selected and developed for input into Maxent. These were created using Geographic Information System processing techniques to make a set of grid files at 100 m cell size for a common study area. These included data on elevation, slope, aspect, water availability in soils, depth to water, depth to restrictive layer, soil texture, geology, distance from intermittent and perennial

streams, distance to playas, distance to ponds, distance to springs, and distance to faults (Table 2). These features were selected to characterize the terrain structure of a rugged environment with mountains over 11,000 feet in elevation located east of the Sierra Nevada Mountains and at the north edge of the Mohave Desert. The approximate 3.4 million acres inside the park boundaries is one of the hottest and driest areas in North America.



VALUE	ROCK TYPE	Area Km2
1	alluvium	5397
2	granodiorite	881
3	sandstone	3366
5	shale	206
6	dolostone (dolomite)	600
7	rhyolite	2064
8	dune sand	89
9	conglomerate	274
10	limestone	542
11	landslide	13
12	water	1
13	felsic volcanic rock	18
14	mudstone	7
15	basalt	4
16	peraluminous granite	32
17	quartz monzonite	119
18	diorite	96
19	plutonic rock (phaneritic)	24
22	andesite	13
23	ash-flow tuff	3
24	quartzite	0
26	gneiss	1
27	playa	0





#### 8 Models - for Historic and Prehistoric sites

#### 1. Historic

a) Residences AH2: Foundations/structure pads, AH4:

Privies/dumps/trash scatters.

b) Mines HP43: Mine structure/building, AH9:

Mines/quarries/tailings.

#### 2. Prehistoric

a) Habitation AP8: Caches, AP7: Architectural features, AP15:

Habitation debris, AP3: Ceramic scatter, AP11: Hearths/pits.

b) Milling AP4: Bedrock milling feature.

c) Rock Art AP6: Pictographs, AP5: Petroglyphs.

d) Rock Shelter AP14: Rock shelter/cave.

e) Cairns and Burials AP9: Burials, AP8: Cairns/rock features

f) Lithic AP12: Quarry, AP2: Lithic scatter.

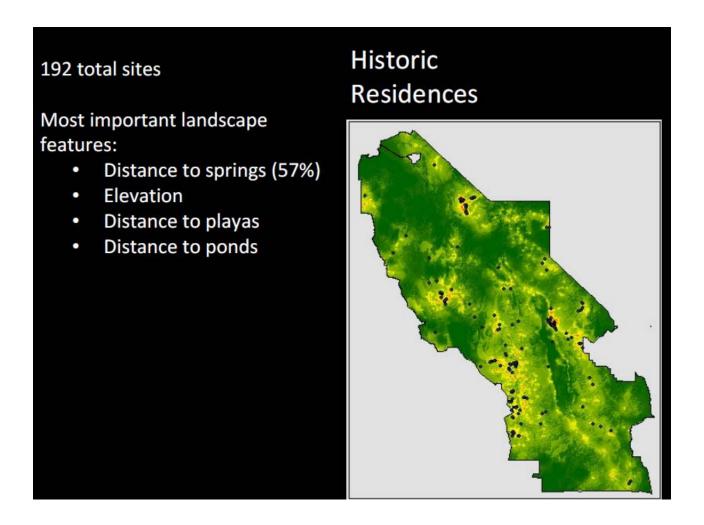
We developed distribution models based on known site locations of eight different types of historic and prehistoric sites.

#### Archeological Sites

The Death Valley region has had various forms of human occupation that date to at least 10,000 years ago, and numerous sites hold evidence of past activities. Among the documented sites, there is evidence of lithic quarries, hunting camps, mines, rock carvings or paintings, and historic residences, among others. The archeological sites identified by the Death Valley park office and provided for this project were coded to reflect the time period and type of use at the site, as well as a number of other features related to the size and survey or report related to the site. For the purpose of this project,

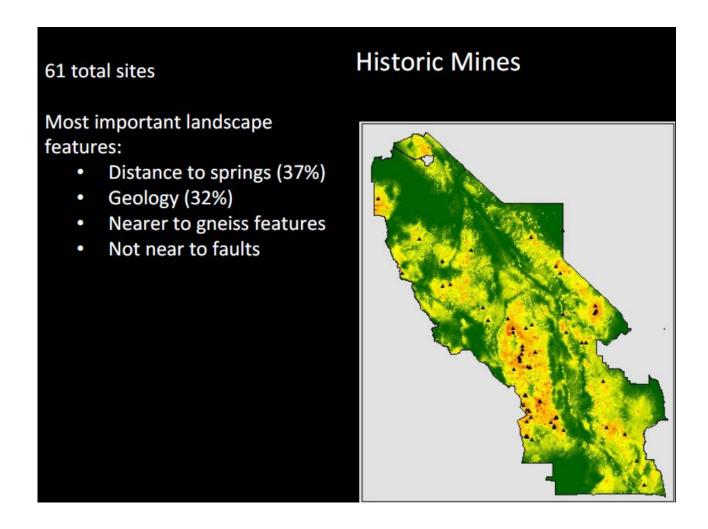
we considered time period distinctions between prehistoric and historic periods. The sites for which models were developed were classified into two historic and six prehistoric site types, as described below.

The most common types of prehistoric site, with at least 200 sites in the group, included the site classes Lithic scatter, Cairns/rock features, Hearth/pits, and Ceramic scatter. Historic sites were much smaller in numbers, with Foundations/structure pads, Mines/quarries/tailings, and Mine structure/buildings being the most numerous. For the purpose of developing models, we ran Maxent with the following classes of sites selected to have a fairly large number of sites and to be representative of site types at the park.



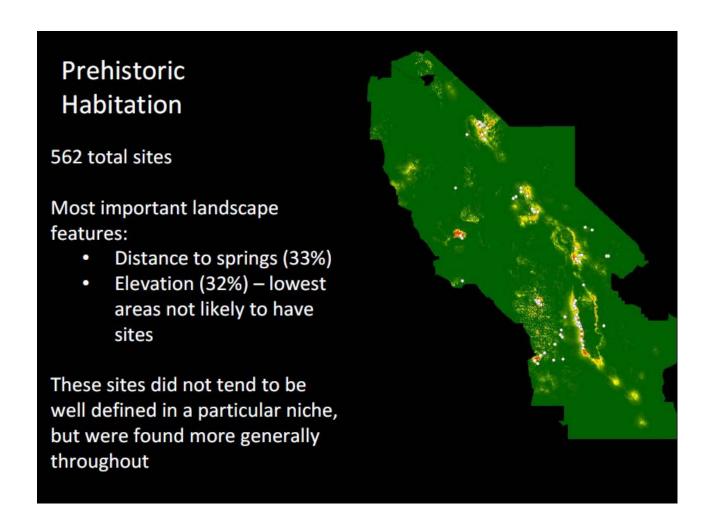
There were 192 total sites used for this model and defined as historic residential sites. The landscape feature most related to the location of these sites was the distance to springs. The distance to springs was the most important variable in all eight of the models, with this one variable contributing to 57 percent of the model outcome in this case. This was

a relatively strong model, with the second highest values in indicators of model power. Elevation was the second strongest variable in the model, while distance to playas and distance to ponds were somewhat important – both provided about a 7 percent contribution.



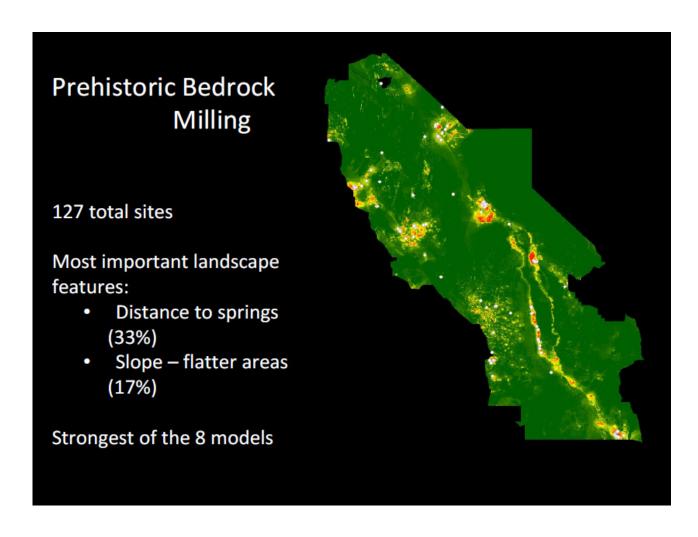
There were 61 total sites used for this model and defined as historic mining sites. The two landscape features most related to the location of these sites were the distance to springs and geology. These two variables were about of equal importance of

37 percent and 32 percent respectively. This was a moderately strong model, with the fourth highest values in indicators of model power. Gneiss features had a strong positive influence on site locations. The sites tended to be away from faults.



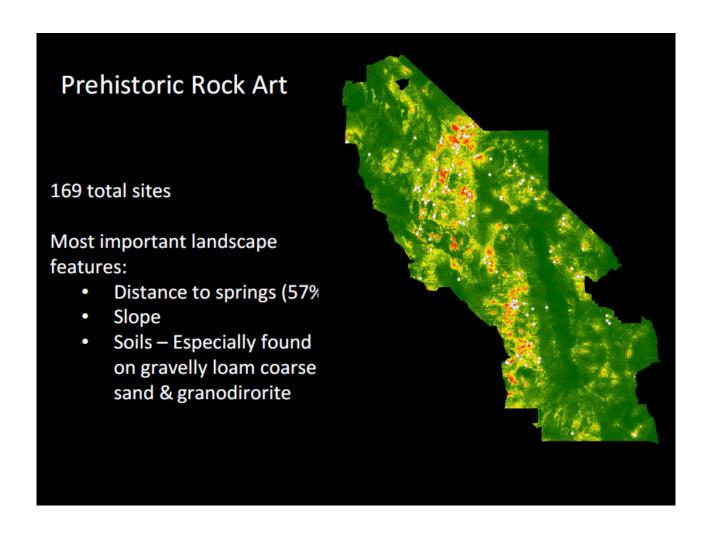
There were 562 total sites used for this model and defined as prehistoric habitation. The two landscape features most related to the location of these sites were the distance to springs and elevation. These two variables were about of equal importance of

33 percent and 32 percent respectively. The lowest elevations were not likely to have evidence of these site types. This model was weaker than all but two other models, probably reflecting the general nature of these sites.



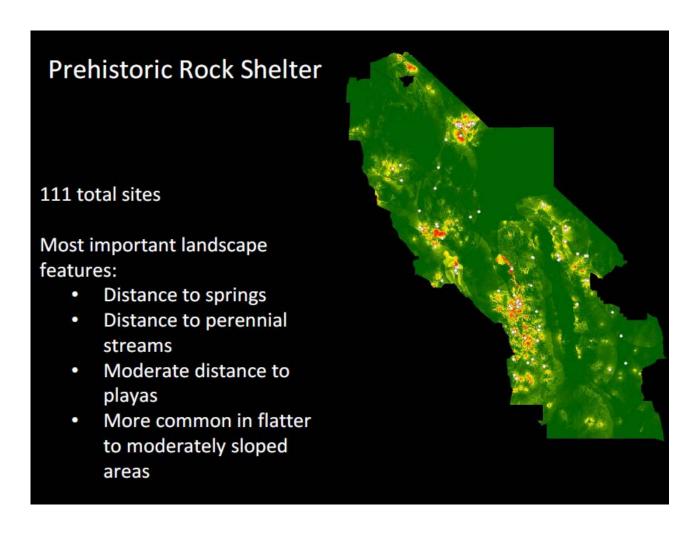
There were 127 total sites used for this model and defined as bedrock milling. In this model, the landscape feature most related to the location of these sites was the distance to springs. The influence of slope was somewhat unusual compared to other

models, with flatter places more likely to support sites. This was the strongest of all of the eight models, indicating that the site definitions and the landscape features included here were well defined.



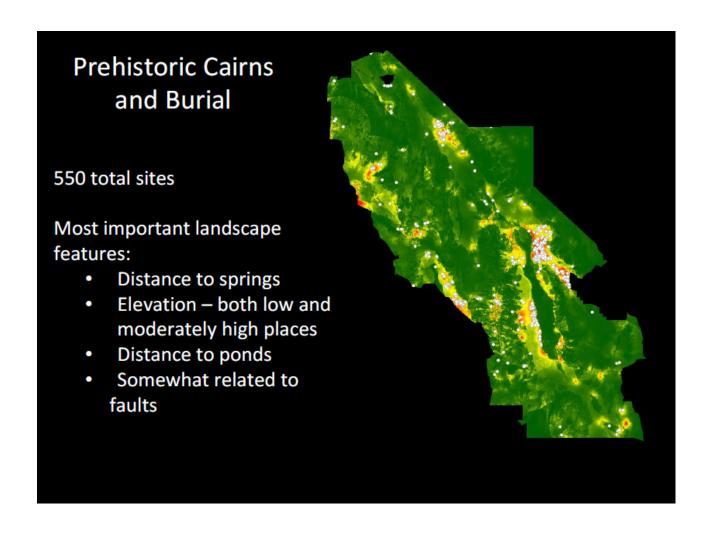
There were 169 total sites used for this model and defined as rock art. Besides distance to springs, this site type also had some influence of slope, with flatter places less likely to be associated with sites. Soils and geology were both important, with gravelly loam coarse sand soil and granodiorite having a positive association and rhyolite have a negative one.

There was tendency for sites to be at a moderate range of elevation, on northwest facing slopes and fewer sites were either at very low and very high elevations. This was the second weakest of the eight models and, with eight of the landscape variables contributing 5 percent or more, was relatively complex.



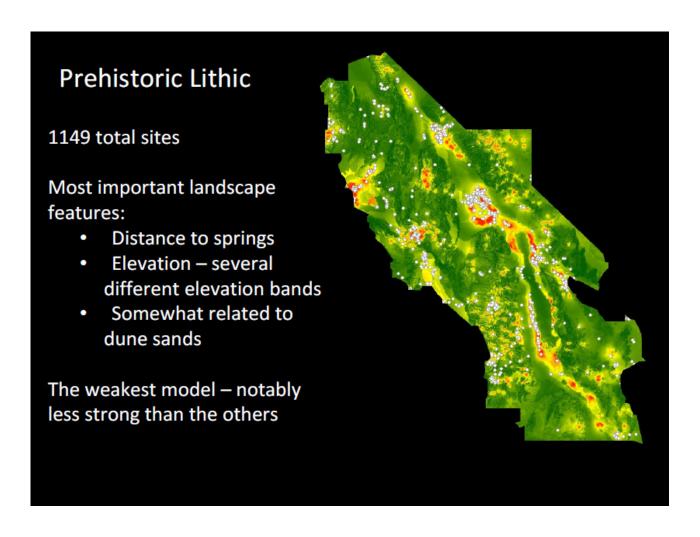
There were 111 total sites used for this model and defined as rock shelters. Besides the importance of location to the distance to springs, three other variables all had a fairly strong influence. These sites tended to be closer to perennial streams, to have

a non-linear relationship to playas, and were more common in flatter to moderate slopes. This was a relatively strong model, the third strongest of the eight.



There were 550 total sites used for this model and defined as cairn or burial sites. Besides the importance of location to the distance to springs, elevation was almost as important at 30 percent and 21 percent respectively. These sites tended to be found at both low and moderately high elevations,

but to not be found in a band of moderate elevation. They were more common closer to ponds and were somewhat associated with faults. This was a moderately strong model, the fifth strongest of the eight.



There were 1149 total sites used for this model and defined as lithic sites. This was the largest site group and represents the most general of the sites, with these sites tending to be found in the same locations as many other site types and not being strongly associated with particular niches. The lithic sites tended to be found near springs, were associated

with several elevation bands, with flatter places, with lower depth to a restrictive layer, and positively associated with dune sand. This was the weakest model of the eight and was notably less strong than the seventh strongest model – prehistoric habitation.

# Summary of model for 8 site types. The AUC value indicates the strength of the model. Numbers in the table are the percentage contribution of that landscape feature

Site Type	Dist Spring	Dist Pond	Dist Playa	Dist Fault	Dist Peren. Strm	Elev	Slope	Aspect	Soil Txt	Soil Dany	Soil Aws	Geol
Residence 192 sites AUC=0.942	57	7	7			8			5			
Mines 61 sites AUC=0.938	37	6		7					6			32
Habitation 562 sites AUC=0.916	33		5			32				6		6
Bedrock Milling 127 sites AUC=0.962	33	6	6		3	6	17		7			
Rock Art 169 sites AUC=0.928	36	6				7	8	5	9		5	9
Rock Shelter 111 sites AUC=0.948	40	5	12		12		10					5
Burials & Cairns 550 sites AUC=0.922	30	9	5	7		21	7					
Lithic 1149 sites AUC=0.866	40					13	8			9		10

#### Discussion and Recommendations

It is important to keep in mind in the interpretation of these models that they were developed and executed as preliminary findings to better demonstrate both the value and challenges inherent in this process. We suggest that several important considerations should guide future modeling of archeological sites at Death Valley National Park:

1. The archeological data present in the DEVA database is biased in multiple and often unknown ways.

Surveying for sites is not often a strictly-managed and scientifically designed effort. Some sites are entered into the database due to casual observations; others were part of specific projects such as road or building construction; and for some sites, the design of the reconnaissance was not recorded or well defined. The tendency to collect site information differentially near roads or other access points could have a particular impact on the types of landscape features that are associated with sites. Different parts of the DEVA have separate management histories as land was added over the years. The focus and specific interests of key individuals during the park's history was especially important in sites entered into the database from older surveys and reports.

A related issue of site data integrity is the precision of the site locations. Some of the older sites were located using small-scale maps and were very imprecise relative to more recent GPS-located sites. The tendency for sites in an area to have similar precision and bias issues would affect the landscape approach used here.

The approach taken here to estimate site potential using Maxent can be adapted to provide an approach that is more sensitive to these biases. It may be that better models could be developed using a smaller number of sites but limiting the set to those with higher data integrity. Another important area that needs to be considered is the selection of the points used by Maxent as the "zero" values against which to compare the site location data. In the models described here, 10,000 random points were created by Maxent to represent the non-site areas. When there are survey results with true zeros, then these can be used instead of random points. In addition, the user can limit the analysis to develop the model only in those areas where surveys have been completed. The need to determine the reliability of the surveys and to develop a more nuanced metric for the site occurrence or lack thereof is an important area of future work.

2. The landscape variables used in the models were selected for use partly due to the resource constraints associated with the project objectives. They were not always ideal as representations of the landscape factors of interest. The geological and soils maps were particularly lacking in detail and were not optimal for identifying important characteristics relative to the site types. The inclusion in the model execution of the full set of all possible options in the soil texture and geological features categorical variables may have made these models more complex than needed. A more judicious approach would be to build the model based on hypothesized relationships, rendered as simplified versions of those data and including only selected options within

them. Future efforts of modeling could be improved by looking at each landscape variable more critically relative to the specific site type in question. If possible, temporal issues could be more clearly represented, with the past landscape features such as vegetation estimates being possible additional environmental layers. Another example would be to estimate the extent to which springs have become dry over time.

- 3. The use of Maxent to develop the models has many appealing characteristics.
- 4. The archeological sites were placed into groups for the development of the eight models, and these groups should be evaluated critically to assess what they represent and to modify these groupings accordingly. For future model development, it might also be useful to create site groups that more likely to have been temporally contemporaneous. Another possibility would be to consider grouping sites as assemblages that have logical connections. It may also be helpful to use the site locations themselves as model input for site location modeling. Sites tend to be near other sites of the same type, for example or there may be a tendency for some sites to be near another type of site. These relationships might be developed from prior knowledge of the staff archeologists, from literature, or developed through spatial data analysis of the existing archeological sites.

In summary, we have found the eco-niche Maxent approach to development of archeological site models for the Death Valley National Park to be informative, but not definitive. We recommend that future models be developed for fewer and more specific site types, taking into account the landscape factors of that are most important. The results of these models can then be interpreted more completely and used to develop maps that reflect management goals and plans, with the understanding

that these models can be run in multiple ways in order to focus on the changing priorities and requirements for sustainable site management.





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# Detecting Mounds Using Airborne LiDAR: Case Studies from Iowa and Minnesota

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#### Discussion and Recommendations

From 2007-2013, Iowa and Minnesota acquired statewide, 1m horizontal resolution Light Detecting and Ranging (LiDAR) imagery, with both states making the data available for unrestricted public download and viewing on the Web. The four studies summarized in this paper found that mounds as small as 3 m diameter and 30 cm high were readily visible in these datasets. The studies successfully identified 37 percent of 8,726 mounds previously recorded at a total of 758 sites located in physiographically-varied regions of each state. Two studies, including one funded by NCPTT, developed LiDAR Surveyor, an ArcGIS model that scans large tracts of land, extracting features with the characteristic 3D geometry of conical mounds in LiDAR. The current version scanned 86 km<sup>2</sup> in seven physiographically-varied areas of interest, identifying 1216 total mound marks (12 km<sup>2</sup>), flagging 88 percent as false positives, and identifying potential mounds at twenty-five of twenty-eight known mound sites within the study areas. The clustering of detected mounds in these 25 groups illustrates the model's utility for prospection, identifying specific areas within which to target costly field verification surveys. The other two studies summarized here achieved mound detection rates of 36 percent by incorporating georeferenced maps and digitized survey traverses to assist in searching LiDAR for 6223 known mounds in seventeen Minnesota counties; 118 previously unknown potential mounds at twelve sites. The studies provided important information about land use factors contributing to mound destruction and preservation. The four studies underscore that archaeologists using LiDAR must be aware of, and explicitly account for, the limitations of LiDAR when using it for archaeological prospection and verification.

#### Introduction

Between 1000 BC and AD 1200, Native Americans in eastern and central North America interred their dead in earthen mounds. Construction of these and other earthworks were part of major changes in the demographic, economic, political, and spiritual organization of human culture throughout the North American continent. The spatial organization of mounds and other earthworks on the landscape has informed scholars about territorial control and astronomical knowledge of ancient people. Archaeological excavations of mounds, although rarely conducted today, provide important insights into prehistoric demography, diet, and pathology, through the osteological analysis of the human interments. In addition to their significance to the humanities, burial mounds are venerated by Native Americans who trace their ancestry to the mounds' builders. Mounds are also among the nation's most threatened archaeological sites. Mounds tend to be concentrated along major rivers and lakes, where urban expansion and recreational development have profound effects on their survival.

Airborne light detection and ranging (LiDAR) has emerged as a technology with great promise for identifying, preserving, and studying ancient earthworks. The LiDAR technology discussed in this paper uses airborne lasers to measure ground surface elevations to submeter accuracy and can yield remarkably images of prehistoric features (Figure 1).

An increasing number of federal, state, and city governments are acquiring LiDAR data for use for a wide variety of purposes. For example, Iowa acquired LiDAR imagery for the entire state between 2007 and 2010, and Minnesota conducted statewide acquisition from 2010-2013

(http://ortho.gis.iastate.edu/#MapLayers; http://www.mngeo.state.mn.us/committee/elevation/mn\_elev\_mapping.html; last accessed 11/18/2015).

The Iowa and Minnesota statewide LiDAR datasets were acquired and processed to USGS standards (Heidemann 2014; Minnesota Geospatial Information Office 2013). Riley (2009) found these accuracy standards to be sufficient to detect mounds as small as 5 m diameter and 30 cm high. The data must achieve a horizontal positional accuracy of 1 m, meaning that 95 percent of the points must be within 1 m of the x,y coordinates assigned to them. Vertical accuracy varies with vegetation cover ranging from 18.5 cm for bare-earth conditions (e.g., a pasture or plowed field) to 37 cm for heavy vegetation. To ensure the highest accuracy possible, 90 percent of the data must be collected under leaf-off conditions.

The Iowa and Minnesota datasets are available for unrestricted viewing and downloading. For archaeologists and the others in the historic preservation community, this poses a double-edged sword. On one hand, it offers the potential of high resolution mapping and spatial analysis of known mounds, and of prospection for unknown features. On the other hand, it increases the vulnerability of mounds to detection and disturbance by vandalism.

#### NCPTT and OSA

Iowa's Office of the State Archaeologist (OSA) is a research unit within the University of Iowa. Its statutory responsibilities include the maintenance of the Iowa Site File, the master inventory of the state's recorded archaeological sites, as well as the protection of all human burials in the state over 150 years in age. NCPTT Preservation Technology and Training (PTT) grants have allowed the office to advance both missions through the application of cutting edge technology.

#### Site Records

In 2003-2005, PTT grant funding allowed OSA to move the Iowa Site File from a desktop GIS to a web-based, interactive map browser called I-Sites (Artz 2003). I-Sites was innovative in several ways, one of which was that it included two separate GIS interfaces to site locations. One, restricted to professional archaeologists, provides a web map

service that allowed users to pan, zoom, and query actual site locations across the entire state. The other, with unrestricted access, displays site counts aggregated by Public Land Survey System sections, displaying information about known site density without revealing exact locations. With occasional improvements, I-Sites has run continuously since its NCPTT-sponsored debut in 2003. I-Sites revolutionized background searches for cultural resource management in Iowa, allowing professional users to query the Iowa Site File from remote computers, without having to travel to Iowa City, as had previously been required (Artz and Eck 2006).

#### **Burials Protection**

Iowa was one of the first states in the nation to develop a positive, forward-looking policy for the treatment of prehistoric Native American human remains that collaboratively addressed Native American concerns for respectful treatment of remains (Anderson et al. 1978). This policy, backed by state law, has successfully allowed OSA to serve as a bastion for the preservation of burial sites over 150 years old, and for the successful negotiation of outcomes where disinterment is unavoidable.

LiDAR Surveyor, OSA's second NCPTT-funded project, was undertaken to assist OSA in that mission by providing a tool for the proactive location of possible ancient burials in advance of threats from development and other vectors of disturbance. PTT Grant MT-2210-11-NC-08 allowed OSA researcher Melanie Riley to continue development of a GIS model, the subject of her master's thesis (Riley 2009), to automate the processing of LiDAR data to search large areas for the characteristic geometry of burial mounds, hopefully leading to the detection and protection of previously unrecorded mound sites.

LiDAR Surveyor runs in ArcGIS, the software used by many if not most agencies, NGOs, and private firms that currently employ GIS technology for historic preservation. The LiDAR Surveyor tool is packed as an ArcGIS toolbox for use in ArcGIS 10 without having to go through an installation process. An operation manual is included that explains how to load the tool into ArcGIS and explains the graphic user interface developed for each step. The tool and manual is available for dissemination to professional archaeologists and

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preservationists with permission from the University of Iowa Office of the State Archaeologist by contacting <a href="http://archaeology.uiowa.edu/contact-us">http://archaeology.uiowa.edu/contact-us</a> (last accessed 11/18/2015).

#### **OSA LiDAR Studies**

The studies discussed here fall into two nonmutually exclusive categories: verification and prospection. Verification studies used LiDAR to locate previously recorded mounds and earthworks at known archaeological sites. These studies not only demonstrate the utility of LiDAR for this purpose, but also served to verify the presence and preservation of mounds at sites that, in some cases, had not been revisited since they were first recorded over 100 years ago. Prospection studies explored methods for automating the processing of LiDAR data to search large areas for previously unrecorded mound sites.

Results of four studies are summarized in this paper. Discussion focuses on results of the studies in terms of the success of mound detection. Methods of analysis are discussed in detail in the individual project reports. The location of AOIs is shown in Figure 2. Projects in Minnesota focused on entire counties, 17 in all. In Iowa, studies focused on particular sites. For simplicity and security, specific sites are not shown; instead, the county containing the sites are shaded.

LiDAR has been employed for prehistoric feature detection in several OSA studies not discussed here. These include Riley (2010, 2012a), Whittaker (2009), Whittaker and Kendall (2007), and Whittaker and Riley (2012).

#### Prospection

LiDAR Surveyor (Riley 2012b) and its prototype (Riley 2009) are examples of a relatively common type of GIS analysis called feature extraction. Feature extraction algorithms scan LiDAR data for patterns that match the geometry of a specific kind of three-dimensional feature, such buildings, and extracts those features into separate datasets for individualized visualization and analysis (Dilts et al. 2010; Hewett 2005; Mass and Vosselman 1999; Opitz et al. 2006; Maune 2007).

#### Mound Geometry

Riley (2009) developed criteria to describe the characteristic geometry shared by burial mounds in Iowa and adjacent states that could be operationalized using common GIS geoprocessing tools. Her data were derived primarily from detailed measurements compiled by Green et al. (2001) for Effigy Mounds National Monument and by Stanley and Stanley (1989) for the Slinde Mound Group, both located in northeast Iowa. These surveys revealed mound diameters ranging from 4.6-16.9 m and 0.35 to 3.63 m in height (Table 1). Although both larger and smaller mounds are known, the metrics in Table 1 provide an accurate impression of the size of conical mounds in Iowa, and were used by Riley (2009) to define a focal neighborhood to define the range of sizes that her algorithms would use to identify conical mounds in LiDAR.

#### Prototype

For the prototype model of what became LiDAR Surveyor, Riley (2009) hypothesized that conical mounds would exhibit facing directions, referred to as aspects, ranging from 0-360 degrees. An abrupt break in slope would occur at the outer margins. Applying concepts techniques from hydrological modeling, she reasoned that a mound could be treated as a watershed with a single pour point at the top of the dome from which water would flow in all directions. Consequently, the total area of accumulated flow as water reached the mound's margins should equal the base area of the mound.

She applied the model to five Iowa study areas, referred to areas of interest (AOIs) distributed across the state in regions of differing physiography (Figure 2). In two areas, in Keokuk and Clinton counties, the location of the mounds being searched for was poorly known and not field verified, making them poor tests for the model's detection capabilities.

For the other three project areas, in Lucas County, at Effigy Mounds National Monument in Allamakee County, and at Slinde Mound State Preserve, the model successfully detected 54 of 72 (75 percent) field-verified mounds that were visible in hillshade relief images of the AOIs (Table 2,

Figure 3a), finding mounds as small as 5 m diameter and 30 cm high. Figure 4 illustrates how a mound group (upper left) was represented in separate maps of aspect and slope, and as marks in the final model output (lower right).

Unexpectedly the model detected not only conical mounds but also linear and effigy mounds. Although not circular in planform, these mound types were similar in width, height, and slope to conicals, and thus passed the model's criteria for marking as possible mounds (Riley 2009).

Despite a high success rate in mound detection, the model unfortunately also marked many non-mound features. In areas that had been thoroughly field-surveyed for mounds, and where mound locations were well documented, 96 percent of all landscape features identified by the model as potential mounds were false positives; in other words, features identified as mounds at locations where field survey had not identified a mound (Table 2, Figure 3). False positives were widely spread throughout the individual AOIs. The map at left in Figure 5, for example, shows the location of all features marked by the prototype model as possible mounds in the Effigy Mounds AOI Figure 5). Manual, visual inspection of off-site mounds in aerial photographs and by visual enhancement identify most upwardly-convex earthen features such as ridges of soils along fence lines and natural levees along stream banks. Other false positives were confirmed by aerial photographs as the canopies of isolated trees or clumps of brush that had escaped filtering during creation of the BE-DEM.

#### LiDAR Surveyor

The second version of Riley's model, the PTT-funded LiDAR Surveyor, was designed to reduce the number of false positives. It was applied to ten AOIs located in different landscape settings in Iowa and Minnesota (Figure 2). As Riley (2012b) ran numerous test runs attempting to find a model that would suffice to identify all potential mounds, it became clear that this was not possible. "One [test] model that does well for low-relief mounds in areas with a lot of noise, either

because of fluvial landscapes or poor vegetation filtration, may not do well with preserving marks for strings of well-formed mounds that are close together on a narrow ridge. Mounds that one [test] model may not have detected were marked heavily by the other model" (Riley 2012b). Therefore, after initial processing steps similar to those of the prototype, the modified-model possible mound marks were further classified according to potential problems that contributed to misclassification of raised features as potential mounds. These problems include mounds that are less regular in shape or that have less pronounced changes in slope at their outer edges. Another process was intended to recognize closely-spaced mounds to prevent their being merge into a single linear feature that might be eliminated because it was too long or subrounded. Another process was applied to large mound-like features that sometimes escaped detection because of their broader, somewhat flattened centers.

In the LiDAR Surveyor model, for all AOIs combined, 1065 marks (88 percent) were false positives, compared to 1148 (96 percent) in LiDAR Surveyor (Table 2), suggesting only a nine percent improvement. However, the prototype scanned only 15.7 km² of land, compared to 86.3 km² scanned by LiDAR Surveyor. Adjusted for survey area, the effectiveness of LiDAR surveyor is seen to be considerably greater, detecting 9 false positives per km², compared to 73 per km² for the prototype (Table 2, Figure 3). The reduction in false positives at Effigy Mounds is apparent in Figure 4, in which all mound-like features detected by the models are shown in yellow.

The reduction in false positives, however, came at a price. LiDAR Surveyor detected only 52 percent (151 of 289 mounds in the seven AOIs, compared to 75 percent (fifty-four of seventy-two) in the prototype study (Table 2; Figure 4). The 23 percent drop in mound detection indicates that LiDAR Surveyor was overly conservative in eliminating mound marks. On the other hand, if it had been used as a prospection tool, the model would

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have detected twenty-five of the twenty-eight sites (89 percent), with the three missed sites having only nine mounds. This gave Riley (2012b) confidence that LiDAR Surveyor had utility as a prospection tool.

Riley (2012b) examined a sample of 134 false positives in the Effigy Mounds and Houston County AOIs. The examination consisted of closer study of LiDAR imagery, supplemented with aerial photography. About 40 percent of the features were modern, manmade structures that did not at all resemble a mound when examined more closely in LiDAR imagery and aerial photographs. These included the semi-conical ends of erosion-control terraces and rural dams, and places where farm buildings had been inadequately cleaned from the LiDAR in creating the bare-earth DEM. Vegetation, natural landscape features, and noise in the DEM itself also contributed to false positives.

Knowledge of the factors that contribute to false positives could be the basis for further improving the LiDAR Surveyor model, or at least as a guide to manually weeding out false positives. However, if used for prospection, the results from LiDAR Surveyor underscore what most archaeologists would take as a given: field-checking is the only way to be sure that a false positive is not actually a mound. LiDAR Surveyor, by reducing the number of false positives, makes this a less daunting task then the prototype model would have indicated.

#### Verification

LiDAR Surveyor was not used in the two studies summarized below for purposes having to do less with the model's efficacy, but with the purpose and scope of the projects. Both studies were aimed, not at prospection for mounds, but verifying known sites. Both began with intensive literature searches to locate the best quality maps and other locational information to determine where mounds had previously been recorded. The LiDAR analysis focused on these specific sites. This differed from the studies discussed in the preceding section, the purpose was to determine the effectiveness of LiDAR for prospecting for mound sites within large swaths of terrain. by scanning large areas.

The intensive processing requirements of LiDAR Surveyor were beyond the needs of a site-specific analysis, where manual visualization techniques were sufficient to meet project goals.

Verification (Riley et al. 2012)

Field verification was the objective of a grantsupported project awarded to UI-OSA by the State Historical Society of Minnesota with funding from that state's Arts and Cultural Heritage Fund (http:// www.legacy.leg.mn/funds/arts-cultural-heritagefund). The project's AOIs were previously recorded mound sites in Scott and Crow Wing counties, Minnesota (Riley et al. 2012). The first task was an extensive review of documents and databases housed at the Minnesota's Office of the State Archaeologist. A preliminary GIS study digitized point features for 791 mounds and other earthworks for which good map data existed. LiDAR analysis detected earthen features on 46 percent (n=37) of the sites, including 279 mounds, four non-mound earthworks, and two house depressions. A total of eighty sites with known mounds were examined in LiDAR. In the process five previously unrecorded possible mound sites were detected. For eighteen sites that could not be detected in LiDAR, previous field surveys or LiDAR indicated total destruction by historic land use.

The major factor in the failure of LiDAR to detect mounds was the poor quality of the LiDAR data. Private sector vendors contracted by Crow Wing County flew the LiDAR missions far too late in the spring. As the work progressed from west to east, data quality progressively decreased as trees leafed out, preventing the laser pulses from reaching the surface (Figure 6). Data for the eastern part of the county was not reliable for mound detection, but unfortunately, most of the mound sites in this county are located near its east boundary, where mortuary sites cluster in the vicinity of several large natural lakes.

In Scott County, the vendor's contract stated only that two-foot contours were to be provided. In level terrain with widely spaced contours, fewer points were needed, and thus these areas were excessively thinned. In steep terrain, a high

density of points was retained in order to produce closer spaced contours. In an example (Figure 7), the vendor thinned data points on a ridgetop, where mounds were located, to as little as 1 per 5-10 m<sup>2</sup>, while the adjacent steep slopes had point densities of 1 per 1.27 m<sup>2</sup>. Consequently, point density on ridge- or terrace-top mound sites was often too low to detect mounds.

The study included a fieldwork component, intended to ground truth the LiDAR results. A sample of ten sites was selected for field visits to identify and map mounds using standard field survey techniques. Because of the poor quality of the LiDAR data in these two counties, the field work ended up detecting more mounds than had been located using LiDAR. The results of the study made clear that before using LiDAR for mound detection, the data and the specifications under which the LiDAR data was obtained and processed must be critically examined for potential errors that might limit the identification of relatively small and subtle earthen features like burial mounds (Riley et al. 2012).

Historic Records Verification (Artz et al. 2013) Again with Minnesota Arts and Cultural Heritage funding administered by the Minnesota State Historical Society, Artz et al. (2013) used a combination of map, site records, and LiDAR analysis to conduct a desktop assessment of all recorded mound sites in sixteen Minnesota counties. The researchers were able to use recently acquired LiDAR obtained to USGS specifications for all of Minnesota. A major purpose of the study was to document that the statewide LiDAR data, recently acquired for the state of Minnesota and flown to USGS specifications, would be of use to Minnesota's Office of the State Archaeologist in identifying and protecting mounds. Unlike the county-acquired data available to Riley et al. (2012), the statewide datasets proved adequate for this purpose.

The study examined 650 mound sites with 7646 individual mounds. Analysis of historical maps and archaeological survey data resulted in the georeferencing of the possible locations of 6223 of these mounds. LiDAR data from these sites were analyzed using a number of methods, including default hillshades, custom hillshades, close-interval

contours, and point clouds of data. A total of 2181 mounded features (28.4 percent) was clearly seen in LiDAR within site areas, and an additional 597 were possibly observed but were too indistinct to be certain, for a total of 2778 mounds (36.2 percent of all documented mounds). Although prospection was not a purpose of the project, 118 possibly new mounds were observed in LiDAR at twelve sites in six counties.

Mound survivorship varied greatly by land use. Fifty-four percent of mounds were found in predominantly wooded areas, but only 7.5 percent of mounds were found in developed areas. Mound survivorship is negatively correlated with the year the mound was first recorded. Only about 5 percent of mounds first recorded in the 19<sup>th</sup> century were identified in LiDAR versus 80 percent of those recorded since 1990. Sites recorded before 1880 average seventeen mounds per site, primarily in large groups, declining to an average of 2.5 mounds per site after 1990.

Unlike the data available to Riley et al. (2012), Artz et al. (2013) which were able to use LiDAR acquired to USGS specifications for the State of Minnesota. One major purpose of the study was to document that the statewide LiDAR data would be of use to Minnesota's Office of the State Archaeologist in identifying and protecting mounds. The data proved more than adequate for this purpose, with none of the severe problems caused by the substandard data available for the earlier study.

### Conclusions

#### Potential Limitations of LiDAR

The statewide LiDAR datasets for Iowa and Minnesota used by Riley (2009, 2012b) and Artz and Riley (2010) were flown and processed to USGS specifications. The data are of sufficient accuracy to detect burial mounds as small as 10 m diameter and 30 cm high. As shown by Riley et al. (2010), caution is imperative if LiDAR has been flown to lesser specifications. This means that archaeologists, and other users of the data, must read the metadata, check the websites serving the data, or as in the case

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of Riley et al. (2012) contact the agency or private sector vendors responsible for its acquisition.

Even good quality LiDAR has caveats with which archaeologists should become familiar. LiDAR is always extensively processed prior to release. Airborne LiDAR is acquired by aircraft flying overlapping passes of the landscape, scanning the terrain with laser pulses that reflect off objects below. All pulses that return to the airplane are measured for their return time, and x. y, z coordinates of the objects off which the pulses bounced are calculated. Within this "total return" cloud of points, a subset reached the ground surface, while others reflect off above ground features such as vegetation and buildings. The three-dimensional total return coordinates are provided to users as LAS files, LAS standing for Laser file format. Although LAS files are often referred to by end-users as the "raw" LiDAR data, they are a subset of the total return, having been extensively thinned to remove redundant data.

Further processing of the LAS data identifies a subset of the total return known as the "last return." These are the points that traveled furthest to reach their targets, which means that they reached and were reflected back from the ground surface. The last return points are used to create bare-earth digital elevation models (BE-DEMs). The studies summarized in this paper make use of 1 m BE-DEMs, and despite stringent accuracy standards, users must understand that the points used in their creation have been sorted and classified to extract non-ground points, then thinned and interpolated from a random scatter of points into a regular grid of elevation values.

In particular, archaeologists must be aware that the processing algorithms that result in the selection of points interpreted as the ground surface are created by nonarchaeologists for nonarchaeological purposes. The emphasis is on the removal of vegetation and of modern structures such as buildings that are primarily rectilinear and straight sided. Conical mounds of earth are not the kinds of features the algorithms are "trained" to look for. Nevertheless, we have shown, the bare-

earth point density is usually sufficiently dense and the algorithms sufficiently robust, that mound-like features are readily detectable, especially in areas where the vegetation canopy is sparse. In wooded and developed areas, however, the BE-DEMs often have a "lumpy" appearance due to unfilterable noise. In such areas, Riley et al. (2010) found that a common occurrence is the planar "scalping" of mound, with upper parts of the mound classified as non-ground points and removed, leaving only a flat-topped "stump" of a mound in the DEM. In such situations, the total return point cloud data often retains the shape of the mound, which can be extracted by reclassification of the LAS data (Riley et al. 2010).

The LiDAR product most often used by archaeologists is the hill-shaded relief models created from BE-DEMs. In the studies summarized herein, most LiDAR-detected mounds were visible in the out-of-the-box hillshades available for download or on-line interactively through web services. It must be kept in mind that the default hillshade used by programs most commonly seen in LiDAR-derived and other hillshaded data is set to mimic a light source from due northwest, 45 degrees above the horizon. This angle may be too high to bring low relief features such as burial mounds into view. At sites on east and south facing slopes, the default northwest light source may cast shadows that obscure, rather than heighten, the relief of prehistoric earthworks.

Two-foot contours produced from LiDAR are also available for downloading for Iowa and Minnesota. While useful for many applications, including the georeferencing of historic maps, their use for mound detection should be avoided. The contours are not generated directly from the 1 m BE-DEMs. Instead, the BE-DEM is simplified to a 3 m resolution by interpolating a value based on interpolation from 3 x 3 m blocks of 1 m cells. Instead of being interpolated from a grid of one elevation value for every 1 m², the contours are interpolated from a grid of one elevation value for every 9 m². Features the height and diameter of burial mounds are easily lost in creation of the two-foot contours. Fortunately, 1 m DEMs are

available for download for both Iowa and Minnesota. Generation of contours and hillshades from the 1 m BE-DEM is strongly recommended for mound detection.

Mounds identified solely based on LiDAR analysis should be field verified. Artz et al. (2013), Riley et al. (2012) and Whittaker and Riley (2012) found that many features that appear mound-like in LiDAR were actually features such as brush piles, bedrock outcrops, straw bales, late historic piles of earth, and false images created in LiDAR.

#### Contours vs. Raster Methods

Riley (2009, 2010) and Riley et al. (2012) used raster methods for mound prospection and verification.

Rasters are grids of cells, each encoded with a value. A BE-DEM is a raster dataset in which each cell has an elevation value. Raster data can be manipulated in the GIS environment to enhance mound relief. The direction and azimuth of the virtual light source can be manipulated to control the contrast of lighted vs. shadows to bring out low relief features. In GIS programs capable of working in 3-D, the view can be rotated and tilted to examine a potential mound from several angles. Vertical exaggeration can also be employed to increase relief to facilitate visualization.

Working with raster data requires advanced skills, intensive computer processing, can be time consuming, and can require expensive extensions such as ESRI's Spatial Analyst and 3-D Analyst. Co-author Bristow found that Interpolating 10 cm contours from 1 m BE-DEMs achieved the same visualization enhancements as hillshade lighting and vertical exaggeration (Artz et al. 2013). Contours at this close an interval far exceeds the vertical accuracy of 1 m LiDAR, but this is acceptable as long as the analyst and end-user understand that the data are not intended to represent actual elevations, but rather to enhance trends in the surface (Timothy Loesch, Minnesota Department of Natural Resources, personal communication 2013).

#### *The Importance of NCPTT*

The empowering effect of NCPTT funding is illustrated by the studies summarized above. Those by Riley et al. (2010) and Artz et al. (2013) were funded for purposes of identifying and verifying important cultural resources, providing

the Minnesota's Office of the State Archaeologist important information for managing and protecting mound sites. In contrast, Riley's (2009) master's thesis was a study conducted in an academic research context, an environment arguably more conducive to developing an innovative feature extraction tool for use with LiDAR. Shortly after completing her thesis and as a GIS specialist at OSA, a PTT grant allowed her to continue her work by creating an improvement of her prototype model.

The PTT grant allowed Riley to escape the constraints of the site-centered, preservation-oriented approach that characterizes most publically-funded archaeological work in the United States. NCPTT not only allowed, but encouraged her to innovate. The proposal solicitation, in fact, explicitly stated that a funded project did not necessarily need to succeed to meet the grant requirements. Failure was an option. This openness to innovate and explore was perhaps the principle reason that OSA was attracted to apply for PTT grants, not only for LiDAR Surveyor, but also for the creation of a web interface for Iowa's archaeological sites inventory.

I-Sites, the result of our first PTT grant (Artz 2003), revolutionized access to archaeological site records in Iowa, and has become indispensable to those doing archaeological research and cultural resource management in the State. In contrast, although available to professional archaeologists at no charge, OSA has received few requests for LiDAR Surveyor. This is due in part to lack of knowledge of the model. Its complexity exceeds the skills of most archaeologists, and the Spatial Analyst software it relies on is not affordable to many in our discipline. Thus, in addition to improvements to the model itself to increase its mound detection capabilities, there is a need to move the model to another open source platform not tied to a single proprietary GIS program.

### Detecting Mounds Using Airborne LiDAR: Case Studies from Iowa and Minnesota

#### References Cited

- Anderson, Duane C., Michael Finnegan, John A. Hotopp, and Alton K. Fisher
  - 1978 The Lewis Central School Site (13PW5):
    A Resolution of Ideological Conflicts at an Archaic Ossuary in Western Iowa. *Plains Anthropologist* 23:183-219.

#### Artz, Joe Alan

2003 *I-Sites: An On-Line Database and GIS*for Iowa Archaeology. Office of the State
Archaeologist, University of Iowa, Iowa
City, Iowa City. Submitted to National
Center for Preservation Technology and
Training, National Park Service, Grant
Agreement Number MT-2210-0-NC-17.

#### Artz, Joe Alan, and Colleen Eck

- 2006 Turning the Corner: Evolution of the I-Sites On-Line Interface. Paper presented at the 64<sup>th</sup> Plains Anthropological Conference, Topeka, Kansas.
- Artz, Joe Alan, Emilia D. Bristow, and William E. Whittaker
  - 2013 Mapping Precontact Burial Mounds in Sixteen Minnesota Counties using Light Detection and Ranging (LiDAR). Contract Completion Report 1976. Office of the State Archaeologist, University of Iowa, Iowa City.
- Dilts, Thomas E., Jian Yang, and Peter J. Weisberg (2010) Mapping Riparian Vegetation with Lidar Data: Predicting Plant Community Distribution Using Height Above River and Flood Height. *ArcUser* (winter 2010:18-21). Electronic document, http://www.esri.com/news/arcuser/0110/files/mapping-with-lidar.pdf, accessed October 13, 2010.
- Green, William, Larry J. Zimmerman, Robin M. Lillie, Dawn Makes Strong Move, and Dawn Sly-Terpstra.
  - 2001 Effigy Mounds National Monument

Cultural Affiliation Report, Volume I. Research Papers 26(3). Office of the State Archaeologist, University of Iowa, Iowa City.

#### Heidemann, Hans Karl

2014 Lidar Base Specification (ver. 1.2, November 2014). In U.S. Geological Survey Techniques and Methods, Book 11, Chap. B4. United States Geological Survey, Washington, D.C. (http://dx.doi.org/10.3133/tm11B4; last accessed 11/23/2015).

#### Hewett, Michael

2005 Automating Feature Extraction with the ArcGIS Spatial Analyst Extension. 25th Annual ESRI International User Conference Proceedings, San Diego. Electronic document, http://www10.giscafe.com/link/display\_detail.php?link\_id=13624, accessed October 13, 2010.

#### Mass, Hans G., and George Vosselman

1999 Two Algorithms for Extracting Building Model from Raw Laser Altimetry Data. ISPRS Journal of Photogrammetry and Remote Sensing 54:153-163.

#### Maune, David F.

2007 DEM User Applications. In *Digital Elevation Model Technologies and Applications: The DEM User's Manual.* 2nd ed. American Society for Photogrammetry and Remote Sensing, Bethesda, Maryland.

#### Minnesota Geospatial Information Office

2012 MN Elevation Mapping Project. Electronic document, <a href="http://www.mngeo.state.mn.us/committee/elevation/mn\_elev\_mapping.">http://www.mngeo.state.mn.us/committee/elevation/mn\_elev\_mapping.</a><a href="http://www.mngeo.state.mn.us/committee/elevation/mn\_elev\_mapping.">httml, accessed 11/23/2015.</a>

Opitz, D.W., R. Rao, and J. S. Blundell

2006 Automated 3-D Feature Extraction from Terrestrial and Airborne Lidar. ISPRS Commission IV: Bridging Remote Sensing and GIS, 1st International Conference on Object-based Image Analysis, Salzburg University, Austria. Electronic document, http://www.commission4.isprs.org, accessed 11/23/15.

#### Riley, Melanie A.

- 2009 Automated Detection of Prehistoric Conical Burial Mounds from LIDAR Bare-Earth Digital Elevation Models. Unpublished Master's thesis, Department of Geology and Geography, Northwest Missouri State University, Maryville.
- 2010 Locating Nebraska Phase Earthlodges with LiDAR Bare-Earth Digital Elevation Models and Return Intensity Data, Mills and Pottawattamie Counties, Iowa. In Cultural Resources of the Loess Hills: A Focus Study to Determine National Significance of Selected Archaeological and Architectural Cultural Resources along the Loess Hills National Scenic Byway, edited by Melody K. Pope, Joseph A. Tiffany, and Angela R. Collins, pp. 5.1 5.27. Contract Completion Report 1700. Office of the State Archaeologist, University of Iowa, Iowa City
- 2012a LiDAR Archaeological Prospection for the McKinney Site (13LA1) and the Immediate Vicinity, Louisa County, Iowa: Site Search 2012061. Copy available from Office of the State Archaeologist, University of Iowa, Iowa City.
- 2012bLiDAR Surveyor: A Tool for Automated Archaeological Feature Extraction from Light Detection and Ranging (LiDAR) Elevation Data. Contract Completion Report 1898. Office of the State Archaeologist, University of Iowa, Iowa City.

- Riley, Melanie A., Joe Alan Artz, William E.
  Whittaker, Robin M. Lillie, and Andrew C.
  Sorensen
  - 2010 Archaeological Prospection for Precontact
    Burial Mounds Using Light Detection and
    Ranging
    (LiDAR) in Scott and Crow Wing Counties,
    Minnesota. Contract Completion Report
    1768. Office of the State Archaeologist,
    University of Iowa, Iowa City.
- Stanley, Lori A., and David G. Stanley
  - 1989 National Register of Historic Places
    Nomination Form: Slinde Mound Group.
    Copy available at Office of the State
    Archaeologist, University of Iowa, Iowa
    City.

#### Whittaker, William E.

2009 Digital Mapping and Ground-Penetrating Radar Survey of the Poisel Mound Group (13DM226, 13DM338), Des Moines County, Iowa. Contract Completion Report 1681. Office of the State Archaeologist, University of Iowa, Iowa City.

#### Whittaker, William E., and Bryan Kendall

2007 Mapping and Assessment of the Hinrichs-Burger Mound Group, 13JH12, Sections 3 and 4, T79N-R6W, Johnson County, Iowa. Contract Completion Report 1521. Office of the State Archaeologist, University of Iowa, Iowa City.

#### Whittaker, William E., and Melanie A. Riley

2012 Human Landscapes in Iowa's Past:
Establishing Mapping Protocols for LiDAR
Identification and Mapping of Prehistoric
Cultural Mound. Contract Completion
Report 1914. Office of the State
Archaeologist, University of Iowa, Iowa
City.

# Detecting Mounds Using Airborne LiDAR: Case Studies from Iowa and Minnesota

### **Tables**

Table 1. Descriptive statistics for Northeast Iowa Mound Groups (from Riley 2009:55).

	Effigy Mounds Nat'l Monument		Slinde Mounds			
	Diameter (m)	Height (cm)	Diameter (m)	Height (cm)		
Median	8.35	55	6.1	95		
Mean	8.96	74.27	7.52	85.87		
Standard Dev.	2.78	54.28	3.39	27.78		
Minimum	5.1	35	4.57	46		
Maximum	16.85	263	15.24	153		

Sources of data: Green et al. (2001); Stanley and Stanley (1989).

Table 2. Model Results.

# PROTOTYPE (Riley 2009)

Area of Interest	Area Scanned (km²)	Number of Re- corded Conicals	Number Detected	% De- tected	Number of False Positives	% False Positives	False Positives per km²
Effigy Mounds	6.7	53	43	81%	428	91%	63.5
Slinde AOI	4.9	17	10	59%	209	95%	42.4
Lucas AOI	4.0	2	1	50%	511	100%	127.8
Total	86.3	72	54	75%	1148	96%	73.3

### **LiDAR SURVEYOR (Riley 2012b)**

Area of Interest	Area Scanned (km²)	Number of Re- corded Conicals	Number Detected	% De- tected	Number of False Positives	% False Positives	False Posi- tives per km²
Effigy Mounds	6.7	58	30	52%	34	53%	5.0
Slinde	4.9	16	5	31%	10	67%	2.0
Lucas	4.0	2	0	0%	97	100%	24.3
Pikes Peak	25.0	122	84	69%	304	78%	12.2
Calhoun 2	2.1	11	0	0%	8	100%	3.8
Blood Run	24.5	58	19	33%	428	96%	17.5
Houston	19.0	22	13	59%	184	93%	9.7
Total	86.3	289	151	52%	1065	88%	12.3

# Figure Captions

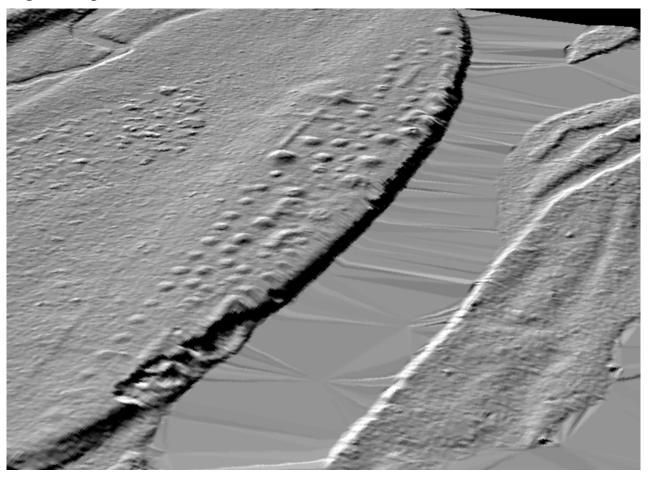


Figure 1. LiDAR image of the Sny-Magill mound group in Clayton County, Iowa (graphic by Melanie Riley).

# Detecting Mounds Using Airborne LiDAR: Case Studies from Iowa and Minnesota

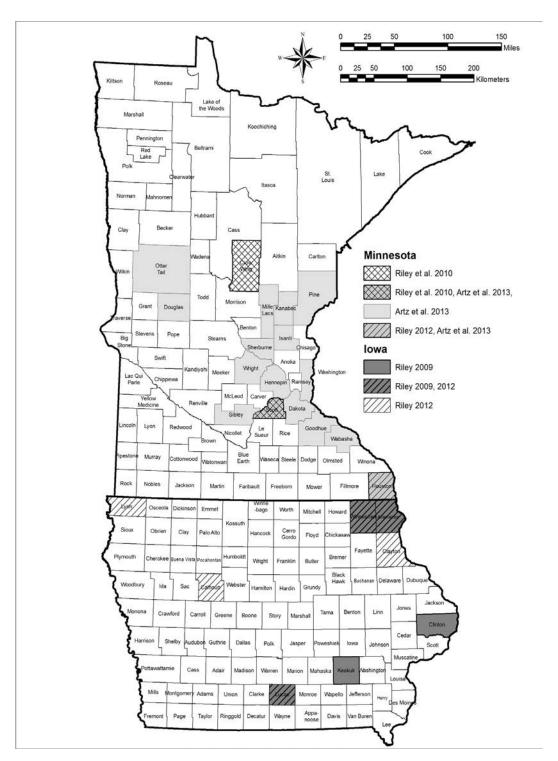
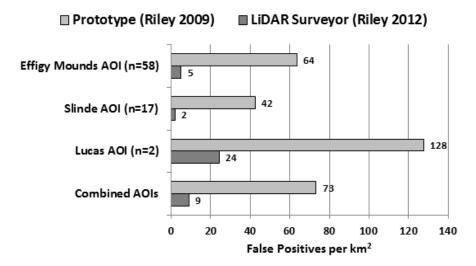


Figure 2. Location of burial mound detection studies conducted by OSA researchers (2008-2012).

### False Positives: Prototype vs. LiDAR Surveyor



### Mound Detection: Prototype vs. LiDAR Surveyor

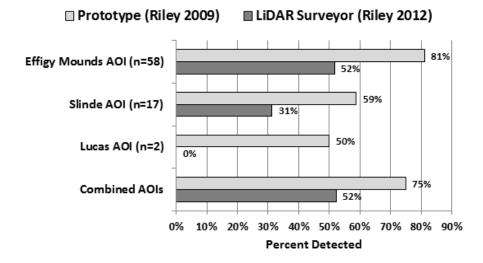


Figure 3. Comparison of prototype and LiDAR Surveyor results. Top: false positive marks per km<sup>2</sup>. Bottom: percent of record mounds detected by models. Recorded mound sample sizes are in parentheses after AOI names.

### Detecting Mounds Using Airborne LiDAR: Case Studies from Iowa and Minnesota

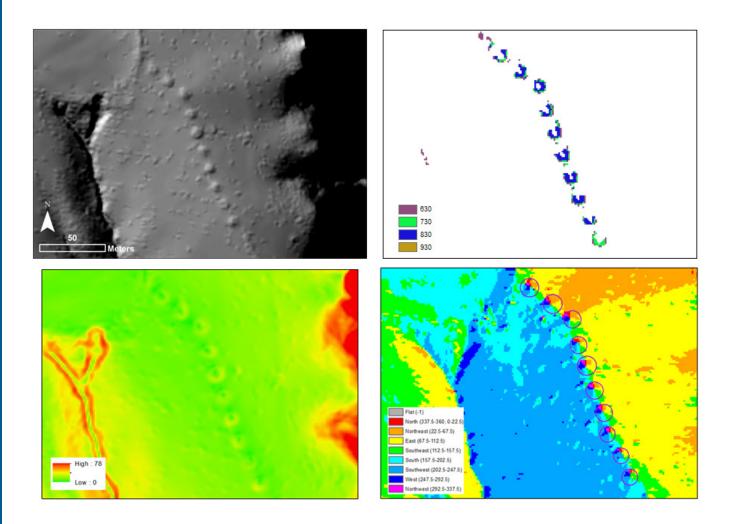


Figure 4. Application of model (graphics from Riley 2012). Upper left: LiDAR hillshade of mound group. Upper right: aspect map of mound group. Lower left: slope map of mound group. Lower right: mound marks identified by model; note also false positives to west of mound group; three digit numbers in legend refer to composite scores assigned by model.

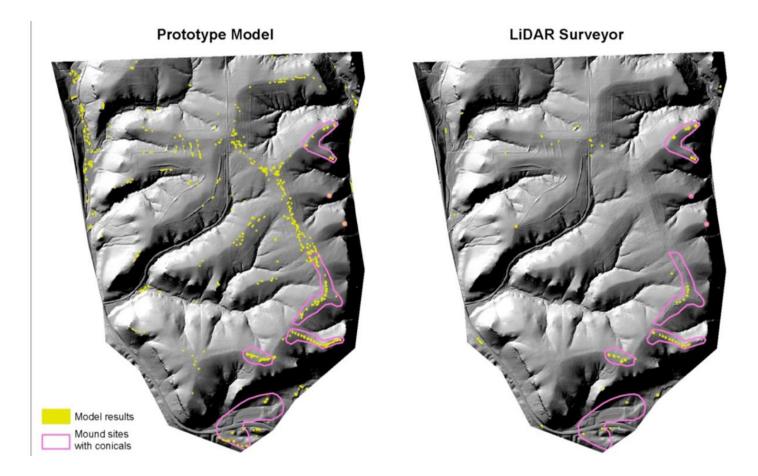


Figure 5. Maps of the Effigy Mounds AOI showing reduction of false positive mound marks in LiDAR Surveyor versus the prototype model.

# Detecting Mounds Using Airborne LiDAR: Case Studies from Iowa and Minnesota

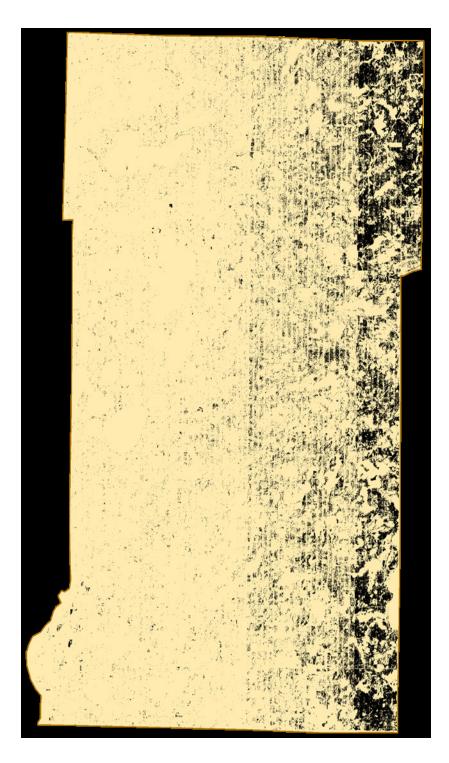


Figure 6. Map of Crow Wing County, Minnesota, showing areas in black that were classified as obscured by vegetation (from Riley et al. 2010).

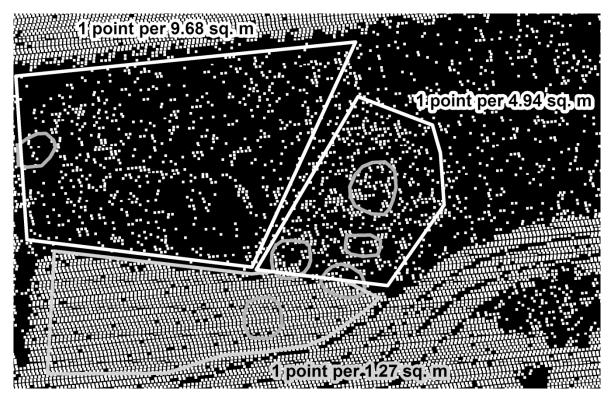


Figure 7. Map of 18CW65 vicinity showing differences in point density in the BE-DEM resulting from the vendor's selective over-thinning of last return points in level versus steep areas (from Riley et al. 2010).

# Incorporating Image Analysis into Ceramic Thin-Section Petrography

### Chandra L. Reedy, University of Delaware

My first experience with digital image analysis of ceramic thin sections was back in 2000, when someone who was then a post-doctoral researcher in my laboratory introduced me to this approach. That was Elizabeth Goins, who is now an associate professor at Rochester Institute of Technology. We explored different approaches to segmenting, or marking, phases for analysis (Goins and Reedy 2000).

Once phases are correctly identified by the software, much information can be quickly obtained about each component. Examples include the overall area percentage occupied by that component; shape characteristics, which may be related to geological source; and size parameters, which may reflect the presence of a temper additive along with silt and other natural components of clay deposits.

I was convinced of the enormous potential of this line of research, but realized that to fully pursue it would require a period of dedicated time and effort. In 2002, our laboratory received a grant from NCPTT to research digital image analysis of petrographic thin sections. That original grant resulted in a document comparing two comprehensive commercial software packages and one free shareware package, taking each through a series of typical operations important for image analysis of archaeological thin sections. Each of these programs works in somewhat different ways,

and each has its own strengths and challenges (Reedy and Kamboj 2003).

*Clemex* is a comprehensive software package, with many capabilities. It was a favorite of a computer science graduate student working in my laboratory. However, I found it to be more difficult to use – I think having some programming background would have helped. *ImageJ* has the big advantage of being free – but we found, at that time at least, it did not have all of the capabilities we needed for our particular uses. For example, we could measure layer thickness at specific points, but could not obtain the average thickness of the entire layer, with standard deviation. Getting technical support when needed was also based upon finding volunteer help posts and wikis. The *Image-Pro* products, by Media Cybernetics, were for me as a non-programmer the most intuitive to use.

When Media Cybernetics came out with their most recent upgrade, *Image-Pro Premier*, I found it to be easy to use and to incorporate everything that I need for image analysis of petrographic thin sections. The company also began to offer free technical support for this product. There are many other programs available – but whichever one a laboratory chooses, there will be the requirement of time and practice to really get good at using it and to be able to use it to full advantage.

The original NCPTT grant work made us competitive for a National Science Foundation grant aimed at further developing protocols for using image analysis with ceramic thin sections (NSF grant 1005992). We began that work with laboratory-prepared specimens. This allowed us to work under controlled circumstances with specimens of known composition. We then began to experiment with various procedures and protocols to segment, or highlight for analysis, particles and pores (Reedy *et al.* 2014a, 2014b).

For particles, we began with the easiest thing to separate for analysis: quartz-rich sand within a ceramic matrix. Once we were comfortable with that, we moved on to more difficult phases. For example, a more difficult problem is how to separate feldspars from quartz. We found that this worked best using plane polarized light images for segmentation, since feldspars tend to look cloudier than quartz. The image analysis program can use that cloudiness as a way of distinguishing between the two phases. However, some additional pre-processing steps were first necessary to clearly distinguish the feldspars from the clay matrix.

Figure 1 demonstrates another more complicated segmentation problem. In this case, the ceramic matrix contained a mica-rich sand. As the upper image in Figure 1 shows, both the clear micas and the clear quartz grains were segmented together by the software, as the colors are close to identical in plane polarized light (and crossed-polarized light cannot be used because it results in many grains being dark and hence invisible). In this case, we were able to use a shape filter to filter out the quartz grains

(as on the lower image in Figure 1) and leave only the micas marked for analysis, since they are more elongated in shape.



Figure 1. When the sand component of a ceramic that is rich in clear micas is segmented by image analysis software, both the clear micas and clear quartz grains get highlighted (upper image). Applying a shape filter leaves only the micas marked for analysis (lower image), since they are more elongated in shape than are the quartz grains.

Twelve years after our initial NCPTT grant, image analysis is now a routine part of thin-section petrography in our laboratory. To show how we incorporate this approach into an overall research program with ceramic thin sections, I will briefly highlight two recent examples.

During the Song dynasty (960-1276 CE) unique black-glazed tea bowls were made at the Jianyang kilns in Fujian Province, China. They were renown throughout China and were also imported into Korea and Japan. Using an iron-rich stoneware body, they had a thick, lustrous glaze, sometimes streaked in black and reddish-brown patterns or silvery colors in what was known as rabbit's hair patterns, or hare's fur glazes (Figure 2, left), or oil droplets, for oil spot glazes (Figure 2, right). Thin-section petrography was done to examine the variability between objects made with the two different glazes (Vandiver and Reedy, 2014).

### Incorporating Image Analysis into Ceramic Thin-Section Petrography

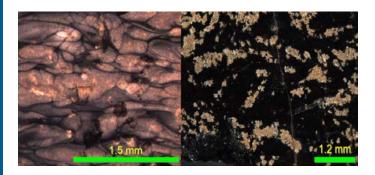


Figure 2. The Jianyang kilns of Fujian Province, China, were renown during the Song Dynasty (960-1276 CE) for unique iron-rich glazes including ones that were described as forming rabbit hair patterns (the 'hare's fur" glazes, left); and ones described as forming rabbit hair patterns (the 'hare's fur" glazes, left); and ones described as looking like oil droplets (the "oil spot" glazes, right).

For quantitative analysis of the silt and sand component, and of pores, both of which are done by image analysis, we started with standard thin sections of 30 microns thickness that were mounted with a blue-dyed epoxy so that pores would be readily visible. For image analysis, we usually use scans of entire thin sections, scanned with a highresolution film scanner that results in a resolution of 5 microns/pixel. This means that analysis of sand particles or pores can be done on the entire thin section, which often contains as many as 15,000 silt and sand grains, and as many as 5,000 pores. Otherwise, even at low magnifications, an image captured under the microscope contains only a very small subset of these. For any analysis of size or shape characteristics, all grains or pores that touch the edges of the image have to be excluded, because they are partially cut off, so their measurements cannot be accurate. Using the entire scanned thin section provides much greater statistical validity to results; otherwise, many separate fields of view would have to be captured under the microscope and their results combined

It is possible with our software to use a tool that allows one to move the thin section across the microscope stage and automatically tile all fields of view into one image. This is useful if the level of detail in a scanned thin section is not at good enough resolution for a particular analysis. However, it is also time-consuming and results in very large image files. For typical projects, the high-resolution film scanner produces a good enough image with which to work.

We then outline and define separate Regions of Interest for analysis, one for the ceramic body and one for the glaze layers (Figure 3). This permits separate image analysis operations to be conducted on the body and on the glaze.



Figure 3. An original scanned image of a Jianyang blackware thin section (mounted in a blue-dyed epoxy) is shown in the upper image. The center image shows the Region of Interest defined for image analysis of the ceramic body, and the lower image the Region of Interest defined for the glaze.

Some of the variables we measured for the ceramic body (Figure 4) are thickness, including mean, minimum, maximum, and standard deviation. Porosity variables measured included Total Optical Porosity (the overall percentage of the ceramic body occupied by visible pores) and the mean,

maximum, and standard deviation for individual pore length, area, perimeter, aspect ratio, roundness, and circularity. The area percentage and these same size and shape characteristics were also measured for the sand component.

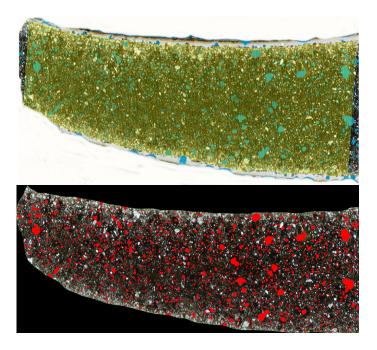


Figure 4. The upper image shows the ceramic body marked for thickness measurements. In the lower image, pores within the ceramic body Region of Interest are highlighted (segmented); data can now be collected on their abundance and their size and shape characteristics.

Image analysis of the scanned thin sections was also used to measure mean, minimum, maximum, and standard deviation of glaze layer thicknesses (Figure 5a) and to measure the maximum bubble size and percent area of the glaze layer comprising bubbles (Figure 5b). Bubbles are often found in Chinese glazes and can affect the optical properties of the glaze.



Figure 5a. Thickness measurements of the glaze layer provide data on the minimum, maximum, and mean thickness, and the standard deviation.



Figure 5b. The Region of Interest marked out for the glaze layer is used to compute the area of the glaze occupied by bubbles, and the maximum bubble size. Bubbles are an important optical component of many Chinese glazes.

The mineralogy of bodies and glazes were then studied qualitatively using traditional thinsection petrography methods under a transmitted light microscope, using the same thin sections that had been scanned. In many cases the glaze has intermittent clumped areas of hematite-rich clay with long, well-developed needles of anorthite that grow from the clay surface. We found that it was helpful to incorporate studies of ultra-thin sections of 15 microns thickness, to better view the smaller crystals that are often seen with high-fired wares. As an example, Figure 6 shows that there is a thin layer of black iron oxide at the glaze surface, red iron-oxide below, and precipitated anorthite needles extending up from the clay boundary, capped with iron-oxide precipitates that indicate the order of crystallization. This formation has been given the aptly descriptive term "waterweeds" (Chen et al. 1986). In another example (Figure 7) there are large radiating clusters of ropy and lath-like anorthite. Precipitated iron oxide feathers out from the edges and fills in the interior spaces of these anorthite structures.

### Incorporating Image Analysis into Ceramic Thin-Section Petrography

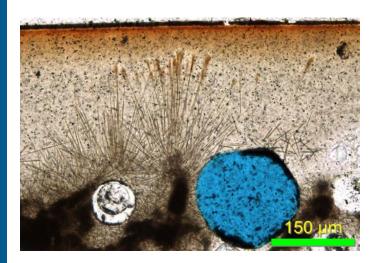


Figure 6. A thin section of a Jianyang blackware sherd shows a thin layer of black iron oxide at the glaze surface with red iron-oxide below. Long anorthite needles extend up from the clay boundary and are capped with iron oxide precipitates; these features are termed "waterweeds."

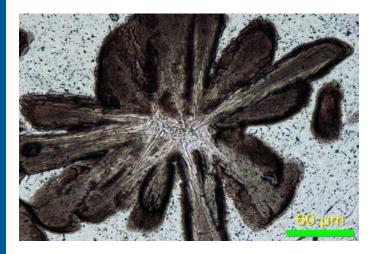


Figure 7. Some Jianyang blackwares have large radiating clusters of ropy and lath-like anorthite within the glaze. Precipitated iron oxide feathers out from the edges and fills the interior spaces of these anorthite structures.\

For documentation of minerals viewed (as with Figures 6 and 7), we always make use of the live extended-depth-of-field feature of the software we use. This creates a very in-focus image even if the thin section is not perfectly ground to the same exact thickness throughout or if there are some phases that are thinner than the section. To use this tool, as you view the image, you turn the microscope in and out of the full range of focus; the software

then automatically produces one very in-focus digital image. This is accomplished using a contrast detection algorithm to analyze the series of images to either extract the one that represents the best-focused image or to create a composite image that takes the best-focused data from the images of the series and combines them into one image.

Statistical analysis included presenceabsence for all qualitative data, as well as the
quantitative data from image analysis. Some
variables indicated a potentially significant
difference between groups, pointing to variables that
may be worth more in-depth study. But variables
showing no significant difference between groups
were just as interesting. For example, percentages
of silt and sand, and size and shape characteristics
of sand grains, are the same for the bodies of both
glaze types (hare's fur and oil spot), as are minerals
related to original raw materials. This tells us that
very similar ceramic bodies were used, no matter
which final product results.

The main differences found relate to firing and cooling regimes. For example, the rosette structures shown in Figure 8 appear in most of the oil spot bodies, but are never found in the hare's fur ones. Forming around a melting quartz particle, the petals and outlines of the rosettes are black magnetite. In between and around the petals, secondary cristobalite has formed.

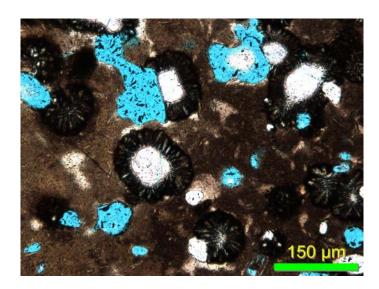


Figure 8. Rosette structures found in the ceramic bodies of Jianyang "oil spot" glazes but absent in the bodies with "hare's fur" glazes are related to firing and cooling regimes. The center is a partially melted quartz particle; the petals and outlines are black magnetite. In between and around the petals, secondary cristobalite has formed.

Most recently, we have been studying ceramics tempered with about 50 percent coal cinders made in

Sichuan Province, China (Reedy *et al.*, 2015). A round, in-ground coal-fired kiln with a shallow pit and

removable ceramic dome cover is used. Pots are pulled out when orange hot and placed with organic material into a deeper covered pit adjacent to the kiln. The organic material burns rapidly and forms a natural ash "glaze" on the exposed surfaces of the pots. The result is a hard black ceramic material with a shiny, silvery, and

sometimes bubbly surface. These porous ceramics resist thermal shock and brittle fracture, making them ideal for their most typical use as soup and stew pots, and hot pots.

As one example of image analysis work on these ceramics, here I highlight the results of image analysis of Total Optical Porosity using the scanned thin sections. Porosity ranges from 11-12 percent for fully fired and glazed soup pots (Figure 9a). Dried but unfired soup pots show only 3 percent porosity (Figure 9b). Most of those pores are small and rounded, appearing to result from chunks of coal cinder popping out during drying. This indicates how firing increases the number, length, and width of elongated pores that reflect pressure applied during fabrication. The rounded pores also enlarge and increase in number during firing. Additional pores develop near the surface within the glassy ash-fluxed glaze layer. For sherds that were fired in the kiln, but did not go into the glaze pit, porosity of the ceramic body is essentially the same as that of the fully fired sherd. The main result of overfiring (Figure 9c) is greatly increased porosity (26 percent Total Optical Porosity), including the appearance of many large bloated pores (some more than 4 mm in length). Some of the increase is due to melting out of mineral phases. Image analysis allows us to quantify these changes and look at the effects of typical firing in the kiln and within the glaze pit.



Figure 9a. The scanned thin section of a coal-clay ceramic soup pot from Sichuan Province, China, shows 12 percent Total Optical Porosity, and one-third of the thin ash-fluxed glaze layer at the top is comprised of pores.

### Incorporating Image Analysis into Ceramic Thin-Section Petrography

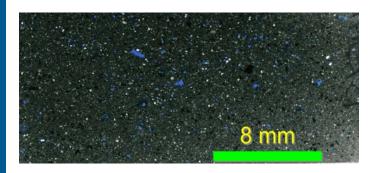


Figure 9b. A dried but unfired coal-clay soup pot shows only 3 percent Total Optical Porosity. Many of those pores are small and rounded, resulting from chunks of coal cinder popping out during drying.

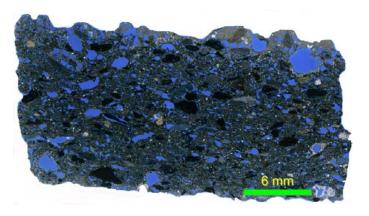


Figure 9c. An overfired coal-clay sherd shows greatly increased porosity (26 percent Total Optical Porosity) with many large bloated pores, some more than 4 mm in length.

To summarize our approach of incorporating image analysis into thin-section petrography of ceramics, it should be emphasized that we do not eliminate qualitative study of minerals under the microscope. Instead, we add to that traditional approach in a number of ways. The new procedures include: (1) use of a dyed epoxy to highlight pores; (2) use of a high-resolution scan of the thin section; (3) use of live tiling to produce thin sections made of many fields of view where necessary; (4) use of live extended-depth-of-field to produce infocus photomicrographs for documentation; (5) outlining a Region of Interest of the ceramic body for segmenting pores and non-plastic particles to collect a full range of data; (6) for glazed ceramics,

outlining a Region of Interest for glaze analysis as well; and (7) including both the quantitative image analysis data and qualitative microscopy data for statistical analyses to address the research questions.

#### References

Chen, Xianqiu; Chen, Shiping; Huang, Ruifu; Zhou, Xuelin; and Ruan, Meiling. 1986. A Scientific Study on Jian Temmoku Wares in the Song Dynasty. In: Shanghai Institute of Ceramics, Academia Sinica, editors. *Scientific and Technological Insights on Ancient Chinese Pottery and Porcelain*. Beijing: Science Press, 227-35.

Goins, Elizabeth and Reedy, Chandra L. 2000. The Application of Image Analysis to Thin-Section Examination in Objects and Architectural Conservation. *Objects Specialty Group Postprints* 17: 122-37.

Reedy, Chandra L. and Kamboj, Sachin. 2003. Comparing Comprehensive Image Analysis Packages: Research with Stone and Ceramic Thin Sections. In: Jane Merritt, editor. *Development of a Web-Accessible Reference Library of Deteriorated Fibers Using Digital Imaging and Image Analysis*. Harpers Ferry, West Virginia: National Park Service, 159-66.

Reedy, Chandra L.; Anderson, Jenifer; Reedy, Terry J.; and Liu, Yimeng. 2014a. Image Analysis in Quantitative Particle Studies of Archaeological Ceramic Thin Sections. *Advances in Archaeological Practice* 2 (4): 252-68.

Reedy, Chandra L.; Anderson, Jenifer; and Reedy, Terry J. 2014b. Quantitative Porosity Studies of Archaeological Ceramics by Petrographic Image Analysis. In: Pamela B. Vandiver, Weidong Li, Christopher Maines, and Philippe Sciau, editors. *Materials Issues in Art and Archaeology X.* Cambridge: Cambridge University Press, doi# 10.1557/opl.2014.711.

Reedy, Chandra L.; Vandiver, Pamela B.; He, Ting; Xu, Ying; and Wang, Yanyu. 2015. Preliminary Investigation of the Coal-Clay Composite Ceramics of Sichuan Province, China. In: 2015 International Symposium on Ancient Ceramics – Its Scientific and Technological Insights. Shanghai: Shanghai Institute of Ceramics, Chinese Academy of Sciences (in press).

Vandiver, Pamela B. and Reedy, Chandra L. 2014. Traditional Craftsmanship and Technology of Jianyang Black Wares from Fujian, China. *Studies in Conservation*, 59 (S1): 169-72.

# Development and Applications of a Minimally Destructive Method of Sourcing Shell via LA-ICP-MS

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### Introduction

Sourcing ceramics via chemical analysis of clays has become a major research area in archaeology over the last few decades. Some perennial problems attend this method and the various techniques commonly employed. Bulk analysis (e.g., via INAA or XRF) can return confusing data due to the incidental inclusion of slips, temper particles, or different clays mixed in ceramic paste recipes, or due to post-depositional elemental alteration of sherd surfaces relative to cores (Schwedt et al. 2004), etc. Techniques employed to ameliorate those problems can bring their own complications (see Larson et al. 2005). The diluting effects of shell temper have been accounted for by mathematical corrections or by chemical removal of temper prior to analysis of clays (Morrow et al. 2003; Steponaitis et al. 1996). Ironically, such steps might be throwing away a sourcing agent of great power.

Some years ago, Peacock et al. (2007) published the results of a pilot study in which ceramic artifacts tempered with freshwater mussel (Unionidae) shell were analyzed for chemical composition of the temper using Laser Ablation-**Inductively Coupled Plasma-Mass Spectrometry** (LA-ICP-MS). This research, which was conducted at the University of California at Long Beach and supported by an NSF grant obtained by Hector Neff to promote the use of LA-ICP-MS in archaeology, was based upon knowledge of mussel behavior and shell growth. Put simply, mussels incorporate trace elements into their calcium carbonate shells in approximate equilibrium with chemical loads in the waterways they inhabit. This characteristic has been widely employed in pollution monitoring studies (e.g., Brown et al. 2005; Das and Jana 2003; Imlay 1982; Markich et al. 2002; Ravera et al. 2003), but until the pilot study of Peacock et al. (2007), it had not been used in sourcing freshwater mussel shelltempered pottery or artifacts, despite the fact that chemical sourcing of marine shell artifacts from North America had been undertaken by that time (e.g., Claassen and Sigmann 1993).

Peacock et al. (2007) analyzed several sherds and a ceramic figurine fragment from the Lyon's Bluff site (22OK520), a Mississippian- to Protohistoric period village in the Black Prairie physiographic province of eastern Mississippi (Peacock and Hogue 2005). Plainwares grouped together chemically, as expected if they were locally produced (see also Morrow et al. 2003, 2-5) using shells from the creek upon which the site is located. Some decorated types fell into distinct chemical groups, while others fell in with the plainwares, suggesting that different source areas were represented (figure 1). Based partly upon the results of this research, a successful major equipment proposal to the National Science Foundation allowed for the acquisition of an LA-ICP-MS system at Mississippi State University (MSU) (Palmer et al. 2004).

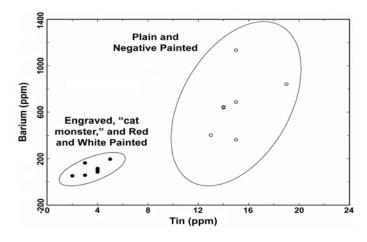


Figure 1. Result of LA-ICP-MS analysis of shell-tempered ceramics from the Lyon's Bluff site, 22OK520 (Peacock et al. 2007, Figure 8).

While the results of this initial project were encouraging, two basic assumptions needed testing before the technique would be ready for widescale adoption. The first was that mussels had been obtained from near the sites where shell accumulated in the past; and 2) chemical differences related to source area would be larger than chemical differences related to other factors: i.e., the provenience postulate would be met. It was recognized that, to be useful for sourcing work, baseline data on shell chemistry must be obtained from archaeological shell specimens, as modern specimens reflect current, post-industrial chemical loads. Some preliminary work had been done by Peacock et al. (2007) using mass digestion and ICP-MS analysis of a small number of archaeological shells from a few different drainages, but more work clearly was needed along these lines. Specifically, work needed to be done using LA-ICP-MS to make the results more directly comparable to what would be obtained from future analyses of shell temper or shell artifacts using this essentially non-destructive approach.

With these needs in mind I submitted a proposal to the National Center for Preservation Technology and Training entitled "Establishing an Elemental Baseline for Sourcing Shell and Shell-Tempered Artifacts in the Eastern Woodlands of North America" (Peacock 2006). Aiding the development of non- or minimally-destructive analytical techniques is a clear part of the NCPTT mission, making the Center a logical choice for a funding request. Under the terms of the proposal, mussel shells from archaeological sites in different states and drainages would be obtained and as many as possible under the proposed budget would be chemically analyzed via LA-ICP-MS. Partnering in the research was Dr. Ronald A. Palmer, at that time a chemist with the Institute for Clean Energy Technology at MSU. The grant was awarded in November 2007 and work was conducted over a 10-month period. In this paper, I briefly recap the results of that work and trace the interconnecting studies that have since arisen as archaeologists have begun to adopt this approach to sourcing of shelltempered ceramics and other artifacts.

### Results of NCPTT-Funded Work

Upon notification that the NCPTT grant had been awarded, two graduate research assistants were employed and a call was put out to the archaeological community seeking donations of shell samples from different areas. Notices were run in the International Council for Archaeozoology Newsletter (Vol. 8, No. 2), the Southeastern Archaeological Conference Newsletter, and other outlets. News of the grant filtered through various media, from local newspapers (e.g., the Columbus Dispatch – http:// www.cdispatch.com/ articles/2007/10/31/local news /area news/; the *Tupelo Daily Journal* - http:// djournal.com/news/shards-of-history/) to a variety of websites (e.g., the Stone Pages Archaeo News [http:// www.stonepages.com/news/archives/002596.html]) and helped bring public attention to the project and the NCPTT's role in supporting preservation-related research. A second call for samples was made in conjunction with a grant from the U.S. Army Corps of Engineers, to build upon those obtained via the NCPTT project in anticipation of conducting isotopic analysis related to paleoclimates in the Lower Mississippi Valley (Peacock 2011). The response from the archaeological community to the request for shell specimens was gratifying: far more samples were obtained than could be analyzed with the resources available, and those samples remain at Mississippi State University, where they are available for other researchers. For example, samples were sent to Lauren Zych, doctoral student at the University of Chicago, for her on-going dissertation research on exploring cultural contact and hybridization through LA-ICP-MS analysis of shell-tempered ceramics from colonial Louisiana (Zych 2013).

As with any new application, various methodological problems arose as analysis got under way, many relating to the vagaries of LA-ICP-MS. Beyond decisions related to analytical parameters such as choice of standards, calibration, laser frequency, beam width, run time, pulse rate, argon carrier gas flow rate, choice of isotopes, etc. (e.g., Bellotto and Miekeley 2000; Fuge et al. 1993), a number of factors can influence the result of any particular analysis. These include things such as surface smoothness of samples, instrumental

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drift, regularity of calibration, etc. In addition, accommodations had to be made for the fact that shell growth varies seasonally, which can affect relative chemical loads. Eventually a useful protocol for shell analysis was worked out, and details are supplied in a number of publications (e.g., Peacock et al. 2007; Peacock et al. 2010). The NCPTTgenerated data are noisier than one would wish, as such protocols were being worked out on the fly. For example, the first data obtained, from shell from a site on the Sunflower River in western Mississippi, comprise a fairly distinct chemical group, but with a great deal of spread in the data (Figure 2). Much of this spread likely is due to the fact that at that point we were not letting the equipment come into thermal equilibrium before taking data. Learning such lessons was a major benefit of NCPTT funding and allowed for improved protocols to be employed in later studies (e.g., Peacock et al. 2014).

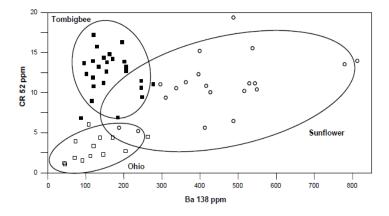


Figure 2. Biplot of shell chemistry from archaeological specimens obtained from different drainages using LA-ICP-MS (Peacock et al. 2012, Figure 2).

As noted by Peacock (2009, 3-4), chemical data were obtained from over a hundred specimens from six sites in three major drainages, including a Middle Archaic shell midden on the Ohio River in Kentucky, a Mississippian site on the Sunflower River in Sunflower County, Mississippi, and four sites in the Tombigbee River drainage of east Mississippi. The Tombigbee assemblages are particularly interesting in that they allow a detailed look into the power of freshwater mussel shell as a sourcing agent. Two of the sites, 22LO527 and

22LO530, are very near to one another on the main stem of the central Tombigbee River and thus should be chemically very similar. The other two sites are on lower-order tributary streams. If analysis showed shells from these individual feeder streams to be chemically distinguishable, it would suggest that the method is potentially even more discriminating than clay sourcing.

Despite the "noisiness" of the data, the results were encouraging. Shells from particular drainages consistently grouped together chemically (Figure 2); i.e., the provenience postulate is being met. This also strongly suggests that shells were obtained from waterways near to the sites and therefore provide a local chemical signature for sourcing studies (see also Peacock et al. 2012). The apparent strength of the method in discriminating sources areas, revealed as protocols improved, is remarkable. For example, shells from sites in the Tombigbee River drainage could readily be sorted into chemical groups via correspondence analysis (Figure 3), with clear discrimination between individual tributary streams and the main river in an area encompassed by a 18-km radius circle (Peacock et al. 2010). This level of spatial resolution rarely is achievable with clay chemistry. In short, the NCPTT-funded work supported the contention that shell chemistry can provide a major line of evidence for sourcing shell-tempered pottery or mussel shell artifacts

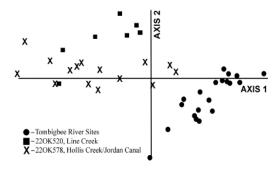


Figure 3. Ordination of chemical data (over 40 elements) obtained from archaeological shells from the Tombigbee River drainage in eastern Mississippi. Sites represented occur within an 18 km-radius circle (Peacock et al. 2012, Figure 3).

### Subsequent Work

If the pilot study reported by Peacock et al. (2007) was the seed, NCPTT funding provided the water, and a number of studies sprouting from these initial efforts will be briefly described here.

Research needs arising from the use of shell in sourcing studies include understanding differences in chemical uptake by species, the chemical effects of burning on shell temper, and the effects of diagenesis on shells in general. Very little work had been done on diagenesis of freshwater mussel shell prior to our sourcing work, but the results were encouraging. For example, Cogswell et al. (1998) found little change in chemical composition of shell as a result of burning (although physical changes in microstructure take place – Maritan et al. 2007); they also found no significant variation between different genera from the same river. In a later study, Peacock and Seltzer (2008) found some difference in calcium and strontium levels in shells of Pleurobema decisum from two strata at the Vaughn Mound site (22LO538) that were widely separated in time. Whether this difference represents the effects of diagenesis or changes in water chemistry over time is currently under investigation (Peacock and Kirkland 2015). A more recent study by Collins (2012) used several methods (e.g., cathodoluminescence, XRD, SEM) to examine shells from deposits of different ages at the Lyon's Bluff site. No evidence of diagenesis (e.g., dissolution of aragonite and reprecipitation as calcite) was noted in this case (Collins 2012).

In 2009, I was contacted by William Parkinson of the Field Museum of Natural History concerning what is referred to as "incrustated" pottery. Such pottery is common at Early Copper Age and later sites on the Great Hungarian Plain, where clay sourcing is difficult due to homogenization of clay beds over large areas. As Parkinson et al. (2010, 66) put it, "The entire eastern half of the Carpathian Basin is filled with fluvially redeposited loess that confounds both petrographic and elemental analyses of ceramic assemblages, making it difficult to differentiate clay sources and patterns of ceramic production and distribution that can be used to model

ancient social interaction." Incrustated pottery has incised lines and/or punctations into which mastic was placed, on top of which a fine, white powder was adhered. Suspecting that the white material was shell, Parkinson requested analysis via LA-ICP-MS of several sherds as a preliminary sourcing study. This analysis, conducted using NCPTT-derived protocols, quickly showed that the materials was not shell but burned and finely crushed bone (Parkinson et al. 2010), a kind of technology known from later sites in the area. This result allowed arguments to be made regarding continuity in ceramic decoration techniques from the Copper Age through the Bronze Age, contradicting earlier scenarios in which local cultures were displaced or subjugated by the socalled Kurgan Invasion (see Parkinson et al. 2010 for details).

One application of chemical analysis is testing whether shellfish were imported to sites for consumption, or whether shell was imported for use as hoes or other artifacts (e.g., Theler 1991). The results of such tests have import for both archaeological questions and resource management considerations. An example of the former comes from Peacock et al. (2010), who tested whether an artifact from the Lyon's Bluff site was an import from the Tombigbee River, minimally some 25 km straight-line distance from the site. The artifact, described in field notes from the 1960s as a "shell spoon," was the only known example of *Lampsilis* straminea claibornensis, a large-river phenotype, to be found at the site, where the small-stream phenotype, Lampsilis straminea straminea, was common in midden deposits. This fact, along with placement in a grave and clear modification (edge grinding) of the shell, suggested that it had been imported to Lyon's Bluff. Chemical testing and comparison with data derived from the NCPTT project indicate that the shell was obtained from Line Creek (figure 4), the waterway adjacent to the site, thus falsifying the hypothesis that it had been imported.

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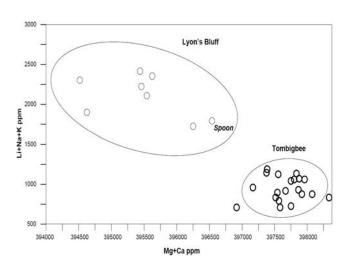


Figure 4. Comparison of chemical data from a shell "spoon" and unmodified freshwater mussel shells from Lyon's Bluff (22OK520) and sites on the main Tombigbee River (Peacock et al. 2010, Figure 4).

In recent years, "applied zooarchaeology" (Lyman and Cannon 2004; Wolverton and Lyman 2001), or the use of zooarchaeological data to provide ecological baselines for modern conservation efforts, has gained a lot of ground. Included in this work are publications on freshwater mussels, which represent one of the most endangered groups of organisms on the planet (e.g., Bogan 2006). One question that proponents of this work face from biologists is how it can be demonstrated that zooarchaeological assemblages represent local faunas rather than imports. As shown with the shell spoon (Peacock et al. 2010), chemical testing is of obvious utility in this regard, and NCPTT-generated data were used in an article to show how such data can be employed as one line of evidence in dealing with questions about the "cultural filter" (Peacock et al. 2012). Further work sourcing mussel-shell tempered

Further work sourcing mussel-shell tempered pottery has been done in the ancestral Caddo region of Texas, Oklahoma, Arkansas and Louisiana by Selden et al. (2014). While they used INAA for their work, they specifically cite Peacock et al. (2007) to argue for focusing on elements known to be common in shell rather than trying to remove them from analysis; i.e., to let the shell chemistry do

the sourcing work. Their study provided far better discrimination of ceramic chemical groups than had been possible with earlier attempts (Selden et al. 2014), bolstering the use of shell as an arguably superior source of data for provenance studies.

Boulanger and Glascock (2015) analyzed 111 valves of freshwater mussel shell, representing several genera, from six sites in the Midwest, the Middle Atlantic, and New England using INAA in order to better understand the chemical contribution of shell temper to ceramic sourcing, finding chemical variation across space and noting that

despite our narrow focus on the effects of freshwater-mussel-shell chemistry on bulk pottery analyses. we reiterate that these data have broader implications beyond viewing shell as a confounding aspect in archaeometric studies. Chemical analyses of shell temper itself may inform directly on selection and procurement of shell by prehistoric potters in the same way in which chemical analyses of ash and sediment may provide greater understanding of ceramic production (Arnold et al., 2000; Hirshman and Ferguson, 2012)... the observation of varying chemical composition among shells [also] strengthens the empirical warrant for attempts to establish provenance of shell artifacts themselves. (Peacock et al., 2010)

### Conclusions

It is fair to say that the tree watered by NCPTT has just begun to sprout (Figure 5). As more work is done showing chemical differences between shells from different drainages, more researchers will become interested in using shell as a sourcing agent, especially if the extraordinary spatial resolution shown by the Tombigbee River drainage data should turn out to be characteristic of what is possible. As a consequence, still further studies into local-scale diagenetic effects will be undertaken. Unlike clay sourcing, where control sampling of the vast North American landscape is in its infancy, freshwater

mussel shell suitable for the generation of chemical background data already exists in huge quantities from drainages of various sizes as a result of previous excavations. This data source grows with work at every shell-bearing site. Collections like the one currently housed at Mississippi State University will allow researchers to make rapid progress in this research area. LA-ICP-MS is a useful method because it is minimally destructive and therefore may be used on rare or culturally sensitive artifacts. But whatever methods are used, the investment by the NCPTT in this area will continue to pay dividends for generations to come.

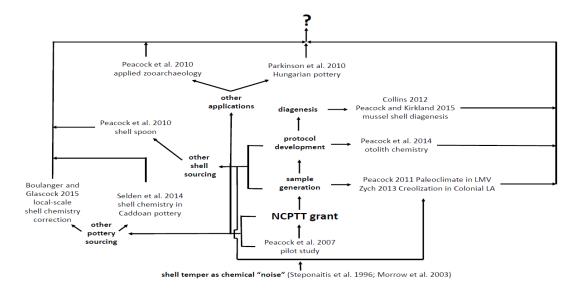


Figure 5. Studies related to chemical sourcing of freshwater mussel shell artifacts, mussel shell-tempered pottery, or mussel-shell diagenesis, and other applications of data or protocols related to NCPTT-funded work on freshwater mussel shell chemistry.

#### Conclusions

First and foremost, I would like to thank David Morgan for his efforts to get my project funded when he worked for the NCPTT. A sincere thanks also to the many archaeologists who contributed shell for the chemical sourcing work. Finally, many thanks to Tad Britt for putting this symposium together. It is an excellent way to recognize the many significant contributions the NCPTT has made, and continues to make, to the discipline of archaeology.

# Development and Applications of a Minimally Destructive Method of Sourcing Shell via LA-ICP-MS

### References Cited

Bellotto, V. R., and N. Miekeley. 2000. Improvements in Calibration Procedures for the Quantitative Determination of Trace Elements in Carbonate Material (Mussel Shells) by Laser Ablation ICP-MS. *Fresenius Journal of Analytical Chemistry* 367:635-40.

### Bogan, Arthur E.

2006. Conservation and Extinction of the Freshwater Molluscan Fauna of North America. In *The Mollusks: A Guide to Their Study, Collection, and Preservation*, edited by C. F. Sturm, T. A. Pearce, and A Valdés, 373-83. Pittsburgh: American Malacological Society.

Boulanger, Matthew T., and Michael D. Glascock. 2015. Elemental Variation in Prehistoric Unionoida Shell: Implications for Ceramic Provenance. *Journal of Archaeological Science Reports* 1:2-7.

Brown, Megan E., Michal Kowalewski, Richard J. Neves, Donald S. Cherry, and Madeline E. Schreiber

2005. Freshwater Mussel Shells as Environmental Chronicles: Geochemical and Taphonomic Signatures of Mercury-Related Extirpations in the North Fork Holston River, Virginia. *Environmental Science and Technology* 39:1455-62.

Claassen, Cheryl, and Samuella Sigmann. 1993. Sourcing Busycon Artifacts of the Eastern United States. *American Antiquity* 58:333-47.

Cogswell, J. W., H. Neff, and M. D. Glascock. 1998. Analysis of Shell-Tempered Pottery Replicates: Implications for Provenance Studies. *American Antiquity* 63:63-72.

#### Collins, J. D.

2012. Assessing Mussel Shell Diagenesis in the Modern Vadose Zone at Lyon's Bluff (22OK520), Northeast Mississippi. *Journal of Archaeological Science* 39:3694-705.

Das, Shamik, and B. B. Jana. 2003. In Situ Cadmium Reclamation by Freshwater Bivalve Lamellidens marginalis from and Industrial Pollutant-Fed River. *Chemosphere* 52:161-73. Fuge, R., T. J. Palmer, N. J. G. Pearce, and W. T. Perkins.

1993. Minor and Trace Element Chemistry of Modern Shells: A Laser Ablation Inductively Coupled Plasma Mass Spectrometry Study. *Applications in Geochemistry* 2: 111-6.

Imlay, Marc J.

1982. Use of Shells of Freshwater Mussels in Monitoring Heavy Metals and Environmental Stresses: A Review. *Malacological Review* 15:1-14.

Larson, Daniel O., Sachiko Sakai, and Hector Neff. 2005. Laser Ablation-Inductively Coupled Plasma-Mass Spectrometry (LA-ICP-MS) as a Bulk Chemical Characterization Technique: Comparison of LA-ICP-MS, Digestion-ICP-MS, and INAA Data on Virgin Branch Anasazi Ceramics. In *Laser Ablation-ICP-MS in Archaeological Research*, edited by Robert J. Speakman and Hector Neff, 95-102. Albuquerque: University of New Mexico Press.

Lyman, R. Lee, and Kenneth P. Cannon (eds.). 2004. *Zooarchaeology and Conservation Biology*. Salt Lake City, Utah: University of Utah Press.

Maritan, L., C. Mazzoli, and I. Freestone. 2007. Modelling Changes in Mollusc Shell Internal Microstructure during Firing: Implications for Temperature Estimation in Shell-Bearing Pottery. *Archaeometry* 49:529-41.

Markich, Scott J., Ross A. Jeffree, and Patrik T. Burke.

2002. Freshwater Bivalve Shells as Archival Indicators of Metal Pollution from a Copper-Uranium Mine in Tropical Northern Australia. *Environmental Science and Technology* 36:821-32.

Morrow, Juliet E., Robert A. Taylor, Robert J. Speakman, and Michael D. Glascock. 2003. Neutron Activation Analysis of Late Mississippian Period Pottery from the Greenbrier Site (3IN1), Independence County, Arkansas. *The Arkansas Archeologist* 44:1-19.

Palmer, Ronald, Adriana Giordana, Feng Han, Evan Peacock, and Jagdish Singh.

2004. Purchase of a Laser Ablation - Inductively Coupled Plasma - Mass Spectrometer (LA-ICP-MS). National Science Foundation Award # CHE 0443643.

Parkinson, W. A., E. Peacock, R. A. Palmer, Y. Xia, B. Carlock, A. Gyucha, R. W. Yerkes, and M. L. Galaty.

2010. Elemental Analysis of Ceramic Incrustation Indicates Long-Term Cultural Continuity in the Prehistoric Carpathian Basin. *Archaeology, Ethnology, & Anthropology of Eurasia* 39 (2): 64-70.

Peacock, Evan.

2006. Establishing an Elemental Baseline for Sourcing Shell and Shell-Tempered Artifacts in the Eastern Woodlands of North America using Laser Ablation – Inductively Coupled Plasma – Mass Spectrometry (LA-ICP-MS). Proposal submitted to the National Center for Preservation Technology & Training, Natchitoches, Louisiana by the Cobb Institute of Archaeology, Mississippi State University, Starkville, Mississippi.

2009. Establishing an Elemental Baseline for Sourcing Shell and Shell-Tempered Artifacts in the Eastern Woodlands of North America using Laser Ablation-Inductively Coupled Plasma-Mass Spectrometry (LA-ICP-MS): Final Report Submitted to the National Park Service, National Center for Preservation Technology and Training for Award # MT-2210-07-NC-08.

2011. Archaeological Freshwater Mussels as Paleoenvironmental Indicators in the Lower Mississippi Valley: Evaluation and Collection of Samples for Analysis. Freshwater Fisheries Investigations Report BAA-EL-11, submitted to the U.S. Army Corps of Engineers, Vicksburg District, by the Department of Anthropology & Middle Eastern Cultures, Mississippi State University, Starkville, Mississippi.

Peacock, Evan, Rinat Gabitov, Jonathan Frisch, Bradley Carlock, and Kate Henderson. 2014. Assessing Site Seasonality and Connectivity via LA-ICP-MS Elemental Analysis of Fish Otoliths: Results of Pilot Study from the Northern Gulf of Mexico. Paper presented at the 71st annual

Southeastern Archaeological Conference, Greenville, South Carolina.

Peacock, Evan, and S. Homes Hogue. 2005. A New Series of Absolute Dates from Lyon's Bluff (22OK520), North Mississippi. *Southeastern Archaeology* 24:46-58.

Peacock, Evan, and Brenda Kirkland. 2015. A Proposal to Investigate Chemical Diagenesis of Freshwater Mussel Shell from the Vaughn Mound Site (22LO538). Proposal submitted to the U.S. Army Corps of Engineers, Mobile District, by Mississippi State University.

Peacock, Evan, Hector Neff, Janet Rafferty, and Thomas Meaker.

2007. Using Laser Ablation-Inductively Coupled Plasma-Mass Spectrometry (LA-ICP-MS) to Source Shell in Shell-Tempered Pottery: A Pilot Study from North Mississippi. *Southeastern Archaeology* 26:319-29.

Peacock, Evan, Ronald A. Palmer, Yunju Xia, Weston Bacon-Schulte, Bradley Carlock, and Jennifer Smith.

2010. Chemical Sourcing of a Prehistoric Freshwater Shell Artifact using Laser Ablation-Inductively Coupled Plasma-Mass Spectrometry. *Archaeology of Eastern North America* 38:91-9.

Peacock, Evan, Charles R. Randklev, Steve Wolverton, Ronald A. Palmer, and Sarah Zaleski. 2012. The "Cultural Filter," Human Transport of Mussel Shell, and the Applied Potential of Zooarchaeological Data. *Ecological Applications* 22:1446-59.

Peacock, Evan, and Jennifer L. Seltzer. 2008. A Comparison of Multiple Proxy Data Sets for Paleoenvironmental Conditions as Derived from Freshwater Bivalve (Unionid) Shell. *Journal of Archaeological Science* 35:2557-65.

Ravera, Oscar, Gian Maria Beone, Robert Cenci, and Paolo Lodigiani.

2003. Metal Concentrations in Unio pictorum mancus (Mollusca, Lamellibranchia) from 12 Northern Italian Lakes in Relation to their Trophic Level. *Journal of Limnology* 62:121-38.

# Development and Applications of a Minimally Destructive Method of Sourcing Shell via LA-ICP-MS

Selden, Robert Z., Jr., Timothy K. Perttula, and David L. Carlson.
2014. INAA and the Provenance of Shell-Tempered Sherds in the Ancestral Caddo Region. *Journal of Archaeological Science* 47:113-20.

Schwedt, A., H. Mommsen, and N. Zacharias. 2004. Post-Depositional Elemental Alterations in Pottery: Neutron Activation Analyses of Surface and Core Samples. *Archaeometry* 46:85-101.

Steponaitis, Vincas P., M. James Blackman, and Hector Neff.

1996. Large-Scale Compositional Patterns in the Chemical Composition of Mississippian Pottery. *American Antiquity* 61:555-572.

Theler, James L.

1991. Aboriginal Utilization of Freshwater Mussels at the Aztalan Site, Wisconsin. In *Beamers, Bobwhites and Blue Points: Tributes to the Career of Paul W. Parmalee*, edited by James R. Purdue, Walter E. Klippel, and Bonnie W. Styles. Illinois State Museum, Springfield, Illinois. Scientific Papers No. 23: 315-32..

Wolverton, Steve, and R. Lee Lyman (eds.). 2012. *Conservation Biology and Applied Zooarchaeology*. Tucson, Arizona: University of Arizona Press.

Zych, Lauren M.

2013. Hybrid Objects and Intercultural Assemblages in Colonial Louisiana. National Science Foundation Dissertation Improvement Grant, Award # BCS-1309751.

# Cold Plasma Oxidation and "Nondestructive" Radiocarbon Sampling

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Mark MacKenzie (Conservation Unit, Museums of New Mexico)

Lukas Wacker (Laboratory of Ion Beam Physics)

The development of radiocarbon dating in the midtwentieth century has revolutionized archaeological chronology (Libby 1955; Wood 2015). The combination of relatively simple theory and the ability to estimate relative isotope abundances resulted in the generation of age estimates for samples of organic carbon from archaeological contexts. In the ensuing decades, the power of radiocarbon dating has increased dramatically. Archaeologists are now much more sophisticated in understanding underlying principles, leveraging that knowledge into a far more reliable selection of samples and interpretation of results. The calibration of atmospheric variation in cosmogenic radiocarbon levels has resulted in much improved calendric date range interpretations, and isotope measurement with accelerator mass spectrometry (AMS) has reduced the size of samples, allowing dating based on annual plant parts.

Even with all of these advancements, a basic limitation has persisted: the perception of radiocarbon dating as a destructive technique. Pretreatments and the transformation of organic compounds into forms of carbon that can be either counted or measured require the destruction of either 10ths of grams or at best milligrams of sample material. Acid-base-acid pretreatment effectively digests sample material during the removal of potential carbonate, oxalate, and humic acid contamination, while graphitization is a common transformative step in preparation of samples for isotope measurement (Aitken 1990; Taylor 1987). Under the conventional approaches available today, the decision to date an object is a decision to sacrifice a tangible part of it to a destructive process.

### Plasma Extraction

In the late 1980s, Marvin Rowe was challenged by colleagues in rock art studies with the problem and potential of dating small amounts of ancient pictograph pigments. The thin-layer applications of organic pigments (charcoal) and the potential use of organic binders in mineral paint layers were outside the realm of the normal approaches to radiocarbon sampling. Amounts of organic carbon in the pigments were extremely small, contamination from carbonates was a risk in many samples, and the amounts of datable carbon that would survive pretreatment were problematic for dating.

In 1989 Rowe was inspired to explore how low-pressure, low-temperature, oxygen plasmas could be used to extract organic carbon from pictograph samples for dating. Jon Russ, Marian Hyman, and Rowe (Figure 1) assembled the first plasma sampling apparatus at the Chemistry Department at Texas A&M University (TAMU), and the first dating sample was produced in 1990 (Russ et al. 1990, 1991). The experimental nature of the venture was emphasized as the radio frequency (RF) generator caught fire and self-destructed at the end of the first sampling run, but the potential of the technique was also confirmed. Over the next several years, three more generations of plasma systems were built (Russ et al. 1993; Chaffee et al. 1993a, b; Ilger et al. 1994b), and additional rock art dates were produced (Hyman and Rowe 1992; Russ et al. 1992a, b; Chaffee et al. 1994a, b, c; Ilger et al. 1994a, b, 1995, 1996).



Figure 1. Marian Hyman, Marvin Rowe, and Jon Russ at the Chemistry Department, Texas A&M University.

A significant advantage of the plasma technique is that the inorganic rock substrate (often including carbonates) does not decompose

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during exposure to low energy oxygen plasmas. This eliminates the need to use extensive acid pretreatments because the plasma temperatures used (< 150°C) are below the decomposition temperatures of both carbonates and oxalate minerals, and only organic carbon is isolated for radiocarbon measurement (Russ et al. 1992b; Chaffee et al. 1993a). Later research added the argument that plasma oxidation is preferable to conventional acid pretreatments because acid washes may not completely remove oxalate minerals, which are commonly associated with rock surfaces and which would contaminate conventional radiocarbon dates (Hedges et al.1998; Armitage et al. 2001).

In 1996, Rowe received a grant from the National Center for Preservation Training and Technology (NCPTT). That funding allowed Rowe and his TAMU students to date further pictograph samples and continue to refine the plasma technique for radiocarbon sampling (Armitage et al. 1997, 1998, 2000b; Hyman and Rowe 1997a, b; David et al. 1999a, 2001; Hyman et al. 1999; Pace et al. 2000; Diaz-Granados et al. 2001; Steelman et al. 2001). By this time the technique was established as arguably the most reliable method for dating pictographs drawn with inorganic pigments: red, yellow, brown, purple and black. The organic matter being dated was presumably due to the addition of binders or vehicles when the paints were made and applied. The NCPTT goal of training new generations of scientists was achieved as TAMU graduate students carried plasma sampling devices with them to their subsequent teaching positions. Professor Karen Steelman has continued this work in her laboratory at the University of Central Arkansas, as has Professor Ruth Ann Armitage at Eastern Michigan University.

Plasma oxidation has successfully dealt with many issues of rock art dating although some concerns still remain (Rowe 2007, 2009; Rowe and Steelman 2003b; Steelman and Rowe 2012). Since its inception, plasma-chemical carbon extraction has been used to date rock paintings from all around the world (Rowe 2001, 2004, 2005a). At least one pictograph has been dated by the plasma oxidation technique in Arizona (Armitage et al. 2000b; Steelman et al. 2004a), California (Armitage et al. 1997, 2005), Colorado (Rowe 2004), Idaho (Steelman

et al. 2002b), Missouri (Diaz-Granados et al. 2001, 2015; Duncan et al 2015), Montana (Chaffee et al. 1994d; Scott et al. 2005), South Dakota (Armitage and Tratebas, unpublished date), Texas (Bates et al. 2015; Boyd et al. 2014; Brock et al. 2006; Chaffee et al. 1993a, b; Hyman and Rowe 1992, 1997 a, b; Hyman et al. 1999; Ilger et al. 1994b, 1996; Jensen et al 2004; Pace et al. 2000; Rowe 2003, 2005b; Russ et al. 1990, 1993), Utah (Chaffee et al. 1993a, b, 1994a, b, c) and Wisconsin (Steelman et al. 2001). The following countries also have pictographs dated by the plasma oxidation technique: Angola (Ilger et al. 1995), Australia (Armitage et al. 1998, 2000a; David et al. 1994, 1997, 1998a, b, 1999a, b, 2000, 2001); Belize (Rowe et al. 2001), Brazil (Rowe and Steelman 2003a; Steelman and Rowe 2005; Steelman et al. 2000, 2002a), France (Ilger et al. 1994a), Guam (Hunter-Anderson et al. 2013), Guatemala (Armitage et al. 2001; Miller et al. 2002; Robinson et al. 2007; Rowe and Steelman 2004, 2007), Mexico (Ilger et al. 1995), Nicaragua (Baker and Armitage 2013), Russia (Steelman et al. 2002c), and Spain (Steelman et al. 2005a).

### Nondestructive Applications

Although Rowe and colleagues were aware of the potential of the plasma sampling technique for nondestructive dating of more than just rock art (Ilger et al. 1996; Hyman and Rowe 1997a, b, 1998), that aspect of the technique was not pursued in earnest until 2002 (Rowe 2005c; Steelman and Rowe 2002, 2004; Steelman et al 2005b; Terry et al 2006). They examined the effect of multiple oxygen plasmas on a shirt tag. The black 'u' shown in Figure 2 faded slightly, but only after many plasma runs (all at the relatively high plasma temperature of circa 150°C). Additional work ensued on the radiocarbon standard TIRI wood sample of Belfast pine as shown in Figure 3. Minor changes can be seen on the thin, almost transparent, right hand side of the sample after the sample was subjected to enough plasma runs to collect more than 20 radiocarbon dates. Dating results for a series of plasma-derived TIRI Belfast pine samples analyzed by Steelman (2004) for her dissertation is presented in Figure 4. Steelman used the early and late results in this series as impetus to explore potential sources of younger and older carbon contamination that could have been introduced as part of the plasma extraction procedure, but the

range of these results is perfectly consistent with the range of both TIRI and FIRI interlaboratory comparison results reported by Scott (2003).

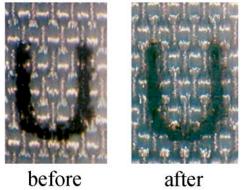


Figure 2. Images of a printed "shirt tag" before sampling and after exposure to many oxygen plasma sampling runs (adapted from Steelman and Rowe 2002).

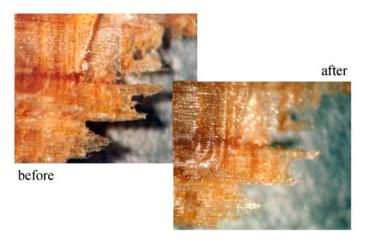


Figure 3. Images of a TIRI wood sample before and after the collection of sufficient carbon for 20 radiocarbon dates (adapted from Steelman and Rowe 2002).

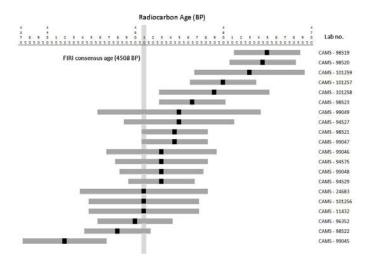


Figure 4. Oxygen plasma derived dates for the TIRI

Belfast pine standard based on samples collected by Karen Steelman for her dissertation research (2004). Radiocarbon assays were by AMS measurement after graphite conversion, and the FIRI consensus age for Belfast pine (4,508 BP) is included for comparison.

A dating project was undertaken on an infant burial recovered from the Lower Pecos River region of Texas (Steelman et al. 2004b). The partially mummified infant had been placed in a grass "nest," with textile wrappings and associated wooden funerary objects. Samples were submitted for three conventional radiocarbon dates, including acid-baseacid pretreatments, and ten plasma CO2 samples were collected. CO<sub>2</sub> samples were collected from a <1 inch square piece of bone and tissue, a blade of grass, a piece of a mat, a piece of twine, and a sotol stalk. An average of the plasma-derived CO, dates was as 2137±13 years BP compared with the average of 2128±20 years BP for the conventional dates. Agreement between dates based on the two sampling approaches is excellent, while visual comparison of the sotol stalk (as an example) before and after three plasma treatments showed no visible change (Figure 5).



Figure 5. Images of a sotol flower stalk before and after radiocarbon sampling (adapted from Steelman et al. 2004b).

### Plasma Extraction

Rowe and colleagues had focused their attention on developing reliable methods of oxygen plasma-extraction of carbon for dating until 2004. The low temperature of the plasma had effectively eliminated concerns about carbonate and oxalate contaminants, however, potential humic acid contaminants remained a concern. To achieve "nondestructive" radiocarbon dating, humic contaminants must be removed (or confirmed absent) without significantly altering the artifact. Conventional practice is to pretreat samples for carbonate and humic acid contamination simultaneously by washing in strong

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acid, then in strong base (alkali), and finally again in strong acid, often at elevated temperatures (usually 50°C). That approach to pretreatment works if the sample is intended for destruction, but the treatment is inconsistent with the potential non-destructive advantages of plasma extraction. Three approaches to humic acid removal have begun to be explored: applications of plasma oxidation itself, pH 8 solution washes, and super critical fluid (SCF) extraction.

Plasma oxidation has been explored successfully as a destructive pretreatment technique (Bird et al. 2010), but there is potential for investigation as a non-destructive pretreatment as well. Destructive pretreatment with oxygen plasmas selectively removed contaminants from crushed charcoal samples at RF power levels of 2 to 100 watts and temperatures up to 150°C. At these elevated temperatures, preliminary acid washes are necessary if carbonate contamination needs to be removed. Organic compounds within the samples are then oxidized differentially, and radiocarbon dating tests of standards revealed that significant proportions of contaminants could be successfully removed. Although low for plasmas in general, the energies and temperatures involved also reduce the total sample volume significantly, ashing up to half the initial sample weight in the process of lowering contaminant concentrations to an acceptable level. Since this oxygen plasma pretreatment is a surface active technique, crushing the sample to maximize particle surface area is necessary in advance of carbon isotope measurement.

The lower temperature plasma conditions used in non-destructive sample collection (described completely below) are much gentler but also appear to have the potential to selectively remove sample components. We have yet to determine how effectively different procedures can be manipulated to remove contaminants, but we have unintentionally been able to distill different fractions of TIRI Belfast pine wood samples at different power and pressure levels of plasma treatment. Experimentation in the potential for differential oxidation of contaminants at low plasma temperatures is an appropriate focus for future research.

Phosphate buffer solutions are another

approach to pretreatment for humic acids that work well with plasma radiocarbon sampling. Mary Ellen Ellis (2008), one of Armitage's students at Eastern Michigan University, used a phosphate buffer solution as a solvent wash for humic acid contaminants. The pH 8.0 buffer solution can remove humic acid contaminants at temperatures of 50°C or less, using repeated ultrasonicated washes and rinses. This pretreatment does require that the object or material be robust enough to soak in the aqueous solution for hours or days at a time, but in combination with the low energy of oxygen plasma sampling, harsh acid-base-acid treatment is not necessary.

In 2004 Rowe received a second grant from NCPTT to support the investigation of SCF extraction as a pretreatment technique. With the NCPTT support, Rowe was able to take sabbatical leave to work with Dr. Jerry King at Los Alamos National Laboratory (LANL). LANL had an SCF group who was willing to collaborate in Rowe's study and explore his idea for removing humic acids by means of SCF dissolution. Unfortunately, LANL access was abruptly closed due to unrelated security breaches, and the proposed study was delayed until the last two weeks of the sabbatical period. The early results were very promising, and King suggested following up this brief initial study. Many factors again delayed the collaboration, and King moved from LANL to become a Professor at the University of Arkansas-Fayetteville. Studies there were first published in 2012 (Lay et al. 2012) followed by a summary by Rowe et al. (2013). SCF has tremendous potential for non-destructive humic acid removal, especially under conditions where the target material could be damaged by exposure to in aqueous solutions.

### The New Mexico Plasma Laboratory

Rowe and the New Mexico coauthors of this paper are currently building a low energy oxygen plasma radiocarbon sampling laboratory at the Center for New Mexico Archaeology (CNMA) in Santa Fe, New Mexico. As a joint venture of the archaeology and conservation divisions of the New Mexico Department of Cultural Affairs, the goal is both to continue research on plasma extraction as a non-

destructive radiocarbon sampling technique and to provide sampling services to the archaeological and museum communities.

The basic architecture of the apparatus is a high vacuum system that is capable of achieving and maintaining vacuums of 10<sup>-6</sup> torr (Figure 6). Glass sample chambers of various sizes are attached to the vacuum systems and to manifolds for the introduction of both high purity argon and oxygen gasses for cleaning and sampling steps. Low energy plasmas are generated at gas pressures of 1-3 torr using an RF generator that can maintain plasmas at power levels as low as 5 watts and chamber temperatures of 35°C or less. After gas samples are generated by plasma oxidation, water vapor is separated with a dry ice-acetone trap, and the CO<sub>2</sub> for radiocarbon dating is condensed within a 4 mm outside diameter glass tube using a liquid nitrogen bath. The glass tube is flame-sealed, retaining the CO<sub>2</sub>, and the ampoule is separated from the apparatus for shipment to the ETH Zurich AMS laboratory under the direction of Lukas Wacker. ETH Zurich is capable of the direct AMS dating of CO<sub>2</sub> samples of 40-100 micrograms, bypassing the need for graphite conversion.



Figure 6. Overview of the plasma sampling apparatus at the Center for New Mexico Archaeology.

While the architecture and theory of sampling are relatively straight forward, the steps in the sampling process are complex and contingent on the characteristics of the materials being sampled. Pretreatment, if necessary, is carried out prior to

initiating plasma sampling. After evacuating the empty sampling chamber to a vacuum of at least 10<sup>-6</sup> torr, research purity oxygen is introduced at a low pressure (1-3 torr). An initial oxygen plasma cleansing of the chamber is then carried out to eliminate any extraneous contaminating carbon from the previous run or from handling of the chamber between runs. Oxygen plasmas are repeated until less than 0.5 micrograms of carbon as carbon dioxide is detected, and then the sample to be processed is placed into the chamber. After the introduction of the sample into the sampling chamber, contamination from modern atmospheric CO<sub>2</sub> must be minimized, both as ambient gas in the system (removed with the high vacuum) and as CO<sub>2</sub> that may be adhering to the surfaces of the sample or the chamber. After evacuating the sample chamber, research purity argon is introduced at low pressure (1-3 torr). The sample is bathed in a low-energy argon plasma (as low as 5 watts and 30°C). Argon is close to CO<sub>2</sub> in molecular size, and the plasma scours the sample and the surfaces of the apparatus, dislodging adhered CO<sub>2</sub>. The sample can off-gas at this stage, releasing water vapor, absorbed CO2 and other gases, and compounds that become volatile under warm low vacuum conditions. Chamber pressure is monitored after each argon plasma run and after the application of liquid nitrogen and dry ice acetone traps, characterizing the amount of evolved or liberated gases. Gases are pumped out of the system, or if there is any reason to retain these gases, those with high enough boiling points can be captured from either of the traps. The volume of gas evolved during cleaning is monitored, and the argon plasma cleaning step is repeated as often as necessary to eliminate the possibility of any significant remaining contamination potential. When less than 0.5 micrograms of carbon as carbon dioxide is captured, the sample is now ready to be processed using the plasma oxidation technique. Since the argon cleaning stage is not chemically reactive, little if any carbon is being removed from the material other than as absorbed or adsorbed CO<sub>2</sub>. The exception may be rare samples whose composition includes hydrocarbon compounds that can be volatilized at the low operating temperature and pressure of the plasma.

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Following the argon cleaning stage, low pressure (1-3 torr) research purity oxygen is introduced. A low energy oxygen plasma is initiated in the chamber (as low as 5 watts and 30°C), and the plasma is maintained for as long as is necessary to produce at least 40-100 micrograms of carbon in the form of  ${\rm CO}_2$ . The time necessary varies with the nature of the material being dated, both in composition and in surface area that is exposed to the plasma. Depending on the sample characteristics, as little as 10 or 15 minutes may be all that is needed to collect enough gas. Chamber size can also affect the amount of carbon that is oxidized irrespective of the amount of time that the plasma is running. Water vapor and traces of other gases are produced in addition to CO<sub>2</sub>. When sufficient CO<sub>2</sub> has been created, the plasma is turned off, and the accumulated gas is subjected first to a liquid nitrogen trap to capture whatever gases have been created in the chamber (primarily  $CO_2$ ). After 10-15 minutes the liquid nitrogen bath is removed and a dry ice acetone trap is initiated in order to separate water vapor and other temperature specific contaminants while releasing the accumulated CO, into the closed system. After determining that sufficient gas has been captured, the gas is subjected to another liquid nitrogen trap to concentrate the CO<sub>2</sub> within a 4 mm outside diameter glass tube. Pressures are monitored to ensure that adequate carbon has been produced, and then the tube is sealed into an ampoule and separated from the apparatus. Multiple samples can be collected as vouchers or for other analyses.

### Research Vignettes

Multiple materials and samples have been subjected to argon and oxygen plasmas during the design and refinement of the CNMA apparatus and sampling procedures. The overall goal has been to build a device and develop protocols that can generate reliable radiocarbon samples with little or no risk of damage to the artifact or material being sampled. This has meant considerable experimentation with electrode design, RF power levels, gas pressures, sampling temperatures, and plasma exposure times, all with a variety of different target materials. The vignettes below represent observations and potential

future research directions for the CNMA laboratory and for plasma radiocarbon sampling in general.

### Plasma Characteristics

Argon and oxygen plasmas have characteristic colors (Figure 7). Those colors change subtly during cleaning and sampling as the gas composition within the sample chamber changes. Color change may provide useful information on the types and compositions of gas mixtures that are created. This may be especially valuable in the oxidation stage of sampling, improving the ability to determine when oxidation has proceeded sufficiently to produce the target volume of CO<sub>2</sub> for dating. More precise determination of this threshold may lower the risk of destructive consequences of sampling on different types of artifacts or materials.



Figure 7. Simultaneous running of two argon plasmas with ring RF electrodes (left) and bar electrodes (back right).

### **Feather Extraction**

A modern turkey feather was subjected to sample collection steps in an effort to explore the effects of the plasma sampling protocol on delicate organic materials. A 'before-plasma' picture was taken of the feather (Figure 8) so that we could assess the extent of physical change due to subsequent plasma exposures. The feather was then inserted into a plasma chamber and subjected to a 1 hour, 5 watt argon plasma. There was no significant release of carbon dioxide, and we began the first of a series of oxygen plasma sampling runs, evacuating the chamber and providing new oxygen at 3 torr for each run. Sampling runs were conducted at 8 watts of RF power for 1 hour; two runs at 10 watts for 1 hour; and a final run at 5 watts for 1 hour. In total, the feather was subjected to 5 plasmas at 5-10 watts of RF power for a total of 5 hours. The feather was removed from the system, examined under 20X for damage, and re-photographed (see Figure 8). No damage was apparent, even to the fine downy

feather components. During the plasma runs, the temperature ranged from 33°C to a maximum of 41.5°C (91.4°F - 107°F).

The feather was again placed into the plasma chamber. It was then subjected to a stronger oxygen plasma of 20 watts RF power for an hour. The maximum temperature on that run was 76°C (169°F). Once again the feather was removed from the system and re-photographed (see Figure 8). At the higher RF power and higher temperature, the feather exhibited visible structural change. The damage was not in the form of erosion of downy components but in an increasing fragility of the structure of the feather and susceptibility to mechanical damage during insertion and withdrawal from the chamber. Even though we did not retain the carbon dioxide from the plasma oxidations, four of the five runs produced more than the 50 to 100 micrograms of carbon (even at 5 watts) required for an accelerator mass spectrometer (AMS) radiocarbon date.

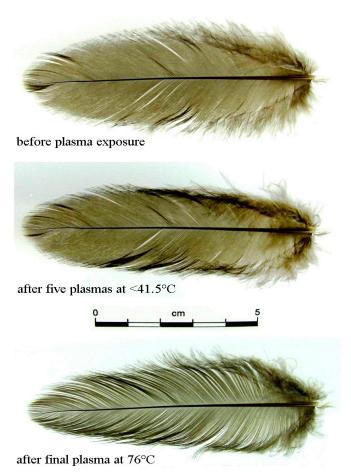


Figure 8. Effects of repeated plasma oxidation sampling on a modern turkey feather. No apparent effect was noted after five sampling runs at RF power of from 5-10 watts and oxygen plasma temperatures of between 33 and 41.5°C (91.4°F - 107°F). A marked fragility is apparent after a single sampling run at 20 watts of RF power and a temperature 76°C (169°F), although fine downy elements appear unaffected.

Our conclusion is that at the lower RF powers, and particularly at 5 watts, with an associated temperature of only about 29°C (84°F) for the argon plasma and 34°C (93°F) for the oxygen plasma, the feather was virtually unchanged visually.

#### Distillation

A variety of RF power settings were used during the collection of calibration radiocarbon samples from a piece of TIRI wood (Belfast pine). Rowe's previous experiences in plasma extraction of TIRI wood radiocarbon samples, extracted at much higher power levels than we currently use, had been unremarkable. However, one combination of settings in the CNMA series resulted in the volatilization

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of a resinous component of the TIRI wood sample (Figure 9). Resin bubbled to the surface of the wood, and a volatile component evolved from the wood and condensed on the interior surface of the sample chamber. The date derived from CO<sub>2</sub> from this run (ETH61251.1) was perfectly consistent with dates on CO, developed from runs that did not fractionate the sample. However, the phenomenon suggests that manipulation of plasma energy and sampling temperature may be used to collect CO, from discrete compositional components of some sample types. This is similar, in a low power sense, to the use of oxygen plasmas as a pretreatment ashing protocol to remove more easily oxidized contaminants prior to collecting CO<sub>2</sub> from more stable components of a sample (Bird et al. 2010).



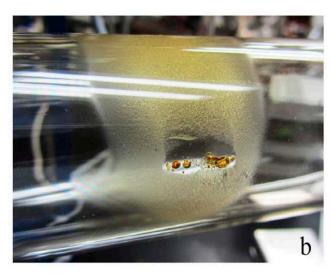


Figure 9. TIRI Belfast pine wood with resinous exudate on the wood surface and condensate on the interior of the sampling chamber tube.

This aspect of the plasma extraction process may also have potential for isolating samples of what actually is contributing to the CO<sub>2</sub> being dated. Gases produced from the material during the initial argon cleaning step can be captured and analyzed by other techniques to provide information on volatile components that might be contributing to the later oxidation step, such as binders in rock art pigments.

### Masking

Composite materials pose challenges to radiocarbon dating, both within and outside the context of non-destructive approaches to sampling. A feature of plasma oxidation is the expectation that only carbon-containing compounds that are directly exposed to energized oxygen species of the plasma will be released from the object being sampled. Exposure to non-energized oxygen molecules should not result in oxidation, and those carbon components should not be included in the radiocarbon sample. This expectation raises the possibility of masking objects to be sampled, allowing only a pre-selected portion to be dated.

A potential application in the Southwestern United States is the radiocarbon dating of the organic paint constituents of potsherds. The organic (carbon) paint is created by preparing and then applying a plant extract to the surface of the unfired vessel, either as a binder for a mineral pigment or by itself within pottery traditions that create designs solely in carbon. During firing, molecules of the plant extract are carbonized within the surface of the vessel, and they should be accessible for plasma oxidation. However, some of the pottery traditions use Cretaceous era carbonaceous clays for vessel construction, and plasma exposure needs to eliminate or minimize contributions of carbon from this source. Two masking approaches will be tried initially, one simply using aluminum foil and the other using a painted-on suspension of inert material (such as aluminum oxide powder). Oxygen species penetrating beneath the foil should lose energy and become non-reactive with carbon compounds that have been protected by the mask. Similarly, oxygen species that diffuse through the porous powder coating should be non-reactive by the time they reach the underlying surface. Both masks

can be removed or reset to allow the collection of radiocarbon samples from different areas of complex artifacts.

If either of these masking approaches is effective, masking will increase the potential applications of non-destructive sampling. For composite artifacts, such as darts, it is conceivable that complementary radiocarbon samples could be submitted from the foreshaft, from sinew attaching the point to the foreshaft, and from any residue adhering to the point.

### Calibration of the New Mexico Apparatus

In May of 2015, a series of calibration samples was submitted to the AMS laboratory at ETH Zurich for radiocarbon dating using a gas ion source for direct insertion of carbon dioxide (Fahrni et al 2013; Ruff et al 2007; Wacker et al 2013). These included CO<sub>2</sub> from the TIRI/FIRI Belfast wood standard (including a sample collected as part of the plasma run with the distillation effect described above, ETH61251.1). Results are presented in figure 10. The FIRI consensus date is 4,508 BP for all measurement methods, while the consensus date for ages estimated by AMS measurement is 4,519 BP (Scott 2003: Table 7.1). The mean of the four New Mexico dates is 4,545 BP, in agreement with the FIRI interlaboratory comparison results. The New Mexico results are also consistent with dates produced from other plasmacollected samples from other laboratories (see Figure 4), including the tendency for mean dates to be slightly older than dates for samples collected and processed by other techniques.

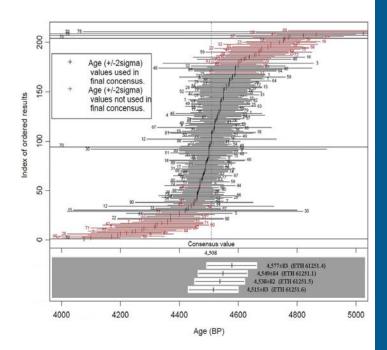


Figure 10. AMS dating results for four New Mexico plasma collections from the TIRI Belfast pine standard (lower panel). The results are compared with the interlab comparison results for the same standard from the FIRI study (adapted from Scott 2003, Figure 7.3).

Risks of contamination in plasma oxidation sampling are ever present due to potential failures of vacuum seals and of argon or oxygen gas contamination. Routine re-sampling of standards, including yet-to-be-sampled dead carbon sources, will be necessary to confirm the reliability of the CNMA sampling technique and the accuracy of the associated radiocarbon dates.

### Conclusions

Plasma oxidation as a radiocarbon sampling technique began as a novel but relatively narrowly focused idea to deal with the challenges of dating rock art. It has evolved into a technique that has the potential to solve not just one but a series of problems in archaeology, museum studies, and conservation. Support from NCPTT was essential in the nurturing and development of the original technique, initiating a research trajectory that continues to expand.

### Acknowledgements

Construction of the plasma radiocarbon sampling lab at CNMA has been supported by the Dr. Don E. Pierce Endowment for Archaeology and

### Cold Plasma Oxidation and "Nondestructive" Radiocarbon Sampling

Conservation, administered by the Museum of New Mexico Foundation.

#### References Cited

Aitken, M.J.

1990. *Science-based Dating in Archaeology*. London and New York: Longman.

Armitage, R.A., J.E. Brady, A. Cobb, J.R. Southon, M.W. Rowe.

2001. Mass spectrometric radiocarbon dates from three rock paintings of known age. *American Antiquity* 66: 471-80.

Armitage, R.A., B. David, M. Hyman, M.W. Rowe, C. Tuniz, E. Lawson, G. Jacobsen, and Q. Hua. 1998. Radiocarbon determinations on Chillagoe rock paintings: Small sample accelerator mass spectrometry. *Records of the Australian Museum* 50: 285-92.

Armitage, R.A., M. Hyman, M.W. Rowe. 2000a. Plasma-chemistry for dating pictographs by AMS. In *Advances in Dating Australian Rock Markings*, edited by G.K. Ward, C. Tuniz, Chapter 4, 31-4. Australia: Australia Rock Art Research Association.

Armitage, R.A., M. Hyman, M.W. Rowe, L.L. Loendorf, and J.R. Southon. 2000b. Dated rock paintings at Red Cliffs, Arizona. *Kiva* 65: 253-66.

Armitage, R.A., M. Hyman, M.W. Rowe, J.R. Southon, and C. Barat. 2005. Fern Cave rock paintings at Lava Beds

National Monument, California: Not the AD 1054
Supernova. In *Current Studies in Archaeoastronomy:*Conversations Across Time and Space, edited by J.
W. Fountain and R. M. Sinclair, Chapter 10, 121-31.
Durham, NC: Carolina Academic Press.

Armitage, R.A., M. Hyman, J.R. Southon, C. Barat, and M.W. Rowe.

1997. Rock-art image in Fern Cave, Lava Beds National Monument, California: not the AD 1054 (Crab Nebula) Supernova. *Antiquity* 71: 715-19.

Baker, S.M., and R.A. Armitage.

2013. Cueva la Conga: First karst cave archaeology in Nicaragua. *Latin American Antiquity* 24: 309-29.

Bates, L. N., A.M. Castaneda, C.E. Boyd, K.L. Steelman.

2015. A black deer at Black Cave: New pictograph radiocarbon date for the Lower Pecos, TX. *To appear in Bulletin of the Texas Archeological Society*, in press.

Bird M.I., P.D.J. Charville-Mort, P.L. Ascough, R. Wood, T. Higham, D. Apperley.

2010. Assessment of oxygen plasma ashing as a pre-treatment for radiocarbon dating. *Quaternary Geochronology* 5: 435-42.

Boyd C.E., M.W. Rowe, and K.L. Steelman. 2014. Revisiting the stylistic classification of a charcoal pictograph in the Lower Pecos. *Bulletin of the Texas Archeological Society* 85: 235-41.

Brock, E., C. Hixson, T. Guilderson, P. Murr, and M.W. Rowe.

2006. Rock painting depicting re-incursion of bison onto the south Texas plains: Painted Indian Cave, Pedernales River, Blanco County, Texas. *Plains Anthropology* 51: 199-205.

Chaffee, S.D., M. Hyman, and M.W. Rowe. 1993a. AMS <sup>14</sup>C dating of rock paintings. In *Time and Space: Dating Considerations in Rock Art Research*, edited by J. Steinbring and A. Watchman, Chapter 7, 67-73. Occasional AURA Publication No. 8. Melbourne: Australian Rock Art Research Association.

1993b. Direct dating of pictographs. *American Indian Rock Art* 19: 23-30.

1994a. Radiocarbon dating of rock paintings. In *New Light on Old Art: Recent Advances in Hunter-Gatherer Rock Art*, edited by D. Whitley and L.L. Loendorf, Chapter 2, 9-12. Institute of Archaeology, Monograph 36, Berkeley/Los Angeles: University of California Press.

1994b. Vandalism of rock art for enhanced photography. *Studies in Conservation* 39: 161-8.

Chaffee, S.D., M. Hyman, M.W. Rowe, N.J. Coulam, A. Schroedl, and K. Hogue.

1994c. Radiocarbon dates on the All American Man pictograph. *American Antiquity* 59: 769-81.

Chaffee, S.D., L.L. Loendorf, M. Hyman, and M.W. Rowe.

1994d. Dating a pictograph in the Pryor Mountains, Montana. *Plains Anthropologist* 39: 195-201.

David, B., R.A. Armitage, M. Hyman, M.W. Rowe and E. Lawson.

1998a. Radiocarbon determinations on Chillagoe rock paintings: small sample accelerator mass spectrometry. *Records of the Australian Museum* 50: 285-92.

1998b. AMS Radiocarbon determinations for north-eastern Australian rock art: Testing the regionalisation model of mid to late Holocene change. In *Congresso International de Arte Rupestre: Atravessando Fronteiras: Congresso Internacional de Arte Rupestre-International Rock Art Congress, 1998*, edited by M.S. de Abreu, CD ROM: file///D|IRAC/ingles/simposios/contents/simp5/ esq\_frames.html. Unidade de Arqueologia da UTAD, Vila Real.

1999a. How old is north Queensland's rockart? A review of the evidence, with new AMS determinations. *Archaeology in Oceania* 34: 103-20.

2001. Landscapes in transition? New radiocarbon dates on cave drawings from the Mitchell-Palmer limestone belt (northeastern Australia). *American Indian Rock Art* 27: 107-16.

David, B., M.W. Rowe, C. Tuniz, and J. Head. 1994. Dating charcoal paintings and drawings from Chillagoe: current research. *Rock Art Research* 11: 127-8.

David, B., C. Tuniz, E. Lawson, Q. Hua, G.E. Jacobsen, J. Head, and M.W. Rowe. 1999b. New AMS determinations for Chillagoe rock art, Australia, and their implications for northern Australian prehistory. In *NEWS '95 - International Rock Art Congress: North, East, West, South, 1995 IRAC. Proceedings*, edited by D. Seglie, 49-56. Pinerolo: Centro Studi e Museo d'Arte Preistorica.

2000. Dating charcoal drawings from Chillagoe, north Queensland. In *Advances In Dating Australian Rock-Markings*, edited by G.K. Ward and C. Tuniz, Occasional AURA Paper No. 10, Melbourne, Australia, 84-9.

David, B., H. Walt, H. Lourandos, M.W. Rowe, J. Brayer, and C. Tuniz.

1997. Ordering the rock paintings of the Mitchell-Palmer limestone zone (Australia) for AMS dating. *The Artefact* 20: 57-72.

Diaz-Granados, C., M.W. Rowe, M. Hyman, J.R. Duncan, J.R. Southon.

2001. AMS radiocarbon dates for charcoal from three Missouri pictographs and their associated iconography. *American Antiquity* 66: 481-92.

2015. AMS radiocarbon dates for charcoal from three pictographs and their associated iconography. In *Picture Cave: Unraveling the mysteries of the Mississippian cosmos*, edited by Diaz-Granados, C., J.R. Duncan and F.K. Reilly III. Chapter 5. Austin: University of Texas Press.

Duncan, J.R., M.W. Rowe, C. Diaz-Granados, K.L. Steelman and T. Guilderson.

2015. The Black Warrior pictograph: Dating and interpretation. In *Picture Cave: Unraveling the mysteries of the Mississippian cosmos*, edited by Diaz-Granados, C., J.R. Duncan and F.K. Reilly III. Chapter 9, 125-31. Austin: University of Texas Press.

Ellis, M.E.

2008. The development of a novel and potentially nondestructive pretreatment for the radiocarbon dating of archaeological artifacts. *Master's Theses and Doctoral Dissertations*. Paper 211, Eastern Michigan University, Ypsilanti, Michigan.

Fahrni, S.M., L. Wacker, H.-A. Synal and S. Szidat. 2013. Improving a gas ion source for <sup>14</sup>C AMS. *Nuclear Instruments and Methods in Physics Research B*, 294: 320-7.

Hedges R.E.M., C.B. Ramsey, G.J. van Klinken, P.B. Petitt, C. Nielsen-Marsh, A. Etchegoyen, J.O. Fernandez Niello, M.T. Boschin, A.M. Llamazares. 1998. Methodological issues in the <sup>14</sup>C dating of rock paintings. *Radiocarbon* 40: 35–44.

### Cold Plasma Oxidation and "Nondestructive" Radiocarbon Sampling

Hunter-Anderson, R.L., M. Yousuf and M.W. Rowe. 2013. Pictographs from Mahlac Cave, Guam: Radiocarbon dating and chemical studies. *American Indian Rock Art* 40: 995-1016.

Hyman, M., and M.W. Rowe. 1992. Radiocarbon Dating of Pictographs. *Texas Archeology* 36(4): 9-11.

1997a. Plasma extraction and AMS <sup>14</sup>C dating of rock paintings. *Techne* 5: 61-70.

1997b. Plasma-chemical extraction and AMS radiocarbon dating of rock paintings. *American Indian Rock Art* 23: 1-9.

1998. Investigating Antiquity: Direct dating of ancient rock paintings. *Science Spectra* 13: 22-7.

Hyman, M., K. Sutherland, M.W. Rowe, R.A. Armitage, and J.R. Southon.
1999. Radiocarbon analyses of rock paintings:
Hueco Tanks, Texas. *Rock Art Research* 16: 75-88.

Ilger, W.A., M., Dauvois, M. Hyman, M. Menu, M.W. Rowe, J. Vezian, and P. Walter.
1994a. Datation radicarbone de deux figures parietales de la grotte du Portel (Commune de Loubens, Ariège). *Prehistoire Ariegeoise* 50: 231-6.

Ilger, W.A., M. Hyman and M.W. Rowe. 1994b. Radiocarbon date for a Red Linear style pictograph. *Bulletin of the Texas Archeological Society* 65: 337-46.

1995. Dating pictographs with radiocarbon. *Radiocarbon* 37: 299-310.

Ilger, W.A., M. Hyman, J. Southon, and M.W. Rowe. 1996. Radiocarbon dating of ancient rock paintings. In *Archaeological Chemistry: Organic, Inorganic, and Biochemical Analysis*, Advances in Chemistry Series, ed. M. V. Orna, Chapter 29, 401-14. Washington, D.C.: American Chemical Society.

Jensen, A., R.J. Mallouf, T. Guilderson, K.L. Steelman, and M.W. Rowe. 2004. Radiocarbon assay and X-ray diffraction analysis of pictograph samples from Tall Rockshelter, Davis Mountains, Texas. *Journal of Big Bend Studies* 16: 31-46.

Lay, J.O., J. Phomakay, K. Srinivas, M.W. Rowe, K.L. Steelman, and J.W. King.

2012. Application of supercritical carbon dioxide – co-solvent mixtures for removal of organic material from archeological artifacts for radiocarbon dating. *International Symposium on a Sustainable Future (ISSF)*, 1-8.

Libby, W.F.

1955. *Radiocarbon Dating*. Chicago: University of Chicago Press.

Miller, A.E., J.E Brady, A. Cobb and M.W. Rowe. 2002. Results of radiocarbon analysis of rock painting from the Cueva de las Pinturas, Guatemala. *Mexicon* 24: 79-81.

Pace, M.F.N., M. Hyman, M.W. Rowe and J.R. Southon.

2000. Chemical pretreatment on plasma-chemical extraction for <sup>14</sup>C dating of Pecos River Genre rock paintings. *American Indian Rock Art* 24: 95-102.

Robinson, E., M. Garnica, R.A. Armitage, and M.W. Rowe.

2007. Los fechamientos del arte rupestre y la arqueología en la Casa de las Golondrinas, San Miguel Dueñas, Sacatepéquez. In *XX Simposio de Investigaciones Arqueológicas en Guatemala 2006*, edited by J. Pedro Laporte, B. Arroyo, and H.E. Mejía, 959-72. Guatemala: Ministerio de Cultura y Deportes, INAH.

Rowe, M.W.

2001. Dating by AMS radiocarbon analysis. In *Handbook of Rock Art Research*, edited by D. Whitley, Chapter 5, 139-66. Walnut Creek: AltaMira Press.

2003. Radiocarbon dating of a deer image from the Lower Pecos River Region, Texas. *Bulletin of the Texas Archeological Society* 74: 83-8.

2004. Radiocarbon dating of ancient pictograms with accelerator mass spectrometry. *Rock Art Research* 21, 145-53.

2005a. Dating studies of prehistoric pictographs in North America. In *Discovering North American Rock Art*, edited by L.L. Loendorf, C. Chippendale and D.S. Whitley, Chapter 12, 294-319. Tucson: University of Arizona Press.

2005b. Radiocarbon dates of charcoal drawings: Hueco Tanks. In *Archaeology of the Jornada Mogollon*, edited by J. Jurgena, 89–94. El Paso: El Paso Archaeological Society.

2005c. Non-Destructive Radiocarbon Dating. Paper #155, 1-13. Art '05 – 8th International Conference on Non-Destructive and Microanalysis for the Diagnostics and Conservation of the Cultural and Environmental Heritage, CD. University of Lecce, Italy.

2007. Reflections on dating rock art. In *Exploring the Mind of Ancient Man*, edited by P.C. Reddy, Chapter 19, 218-31. New Delhi, India: Research India Press.

2009. Radiocarbon dating of ancient rock paintings. *Analytical Chemistry* 81: 1728-35.

Rowe, M.W., A.B. Cobb, P.A. Peterson, and P. McAnany.

2001. Late Classic pictographs from Actun Ik. In *Sacred Landscape and Settlement in the Sibun River Valley*, edited by P.A. McAnany, Chapter 6, 79-85. Boston: Boston University Press.

Rowe, M.W., J. Phomakay, J.O. Lay, O. Guevara, K. Srinivas, W.K. Hollis, K.L. Steelman, T.W. Stafford, Jr., S.L. Chapman, and J.W. King. 2013. Application of supercritical carbon dioxide co-solvent mixtures for removal of organic material from archaeological artifacts for radiocarbon dating. *Journal of Supercritical Fluids* 79: 314-23.

Rowe, M.W., and K.L. Steelman. 2003a. Comment on "Some evidence of a date of first humans to arrive in Brazil." *Journal of Archaeological Science* 30: 1349-51.

2003b. Dating rock art: An imperative need for independent verification. *Mammoth Trumpet* 18(2): 4-7,14,15.

2004. El 'Diablo Rojo' de Amatitlán: aplicación de una técnica no destructiva de cronología por

radiocarbono. In *XVII Simposio de Investigaciones Arqueológicas en Guatemala 2003*, edited by J. P. Laporte, B. Arroyo, H.L. Escobedo, and H.E. Mejía, 1085–97. Ministerio de Cultura y Deportes, Instituto de Antropología e Historia, Asociación Tikal, Guatemala City.

2007. Fechamiento del "diablo rojo" de Amatitlán. Fechamiento radiocarbóno no destructivo. *Rupestreweb* 13: 1-16.

Ruff, M., L. Wacker, H.W. Gäggeler, M. Suter, H.-A. Synal and S. Szidat. 2007. A gas ion source for radiocarbon measurements at 200 kV. *Radiocarbon*, 49: 307-14.

Russ, J., M. Hyman, and M.W. Rowe. 1992a. Dating and chemical analysis of Pecos River Style pictographs. *American Indian Rock Art* 18: 35-42.

1992b. Direct radiocarbon dating of rock art. *Radiocarbon* 34: 867-72.

1993. Direct radiocarbon dating and chemical analysis of ancient rock paintings. *Archaeology and Natural Science* 1: 127-42.

Russ, J., Hyman, M., Shafer, H.J., and M.W. Rowe. 1990. Radiocarbon dating of prehistoric rock paintings by selective oxidation of organic carbon. *Nature* 348: 710-1.

1991. <sup>14</sup>C dating of ancient rock art: A new application of plasma chemistry. *Plasma Chemistry and Plasma Processing* 11: 515-27.

Scott, E.M.

2003. The third international radiocarbon comparison (TIRI) and the fourth international radiocarbon comparison (FIRI), 1990-2002: Results, analyses, and conclusions. *Radiocarbon* 45: 135-408.

Scott, S.A., C.M. Davis, K.L. Steelman, M.W. Rowe, and T. Guilderson.

2005. Dates from four late prehistoric rock art sites in west central Montana. *Plains Anthropologist* 50: 57-71.

### Cold Plasma Oxidation and "Nondestructive" Radiocarbon Sampling

Steelman, K.L.

2004. Non-destructive radiocarbon and stable isotopic analyses of archaeological materials using plasma oxidation. *Ph.D. Dissertation in Chemistry*. Texas A&M University, College Station, Texas.

Steelman, K.L., F. Carrera Ramírez, R. Fábregas Valcarce, T. Guilderson, and M.W. Rowe. 2005a. Direct radiocarbon dating of megalithic paints from north-west Iberia. *Antiquity* 79: 379-89.

Steelman, K.L., J.P. Childress, J. Kolber, M.W. Rowe, and T. Guilderson.

2004a. San Pedro Eye of the Cave: Painting of the past dated for the present. *American Indian Rock Art* 30: 119-28.

Steelman, K.L., R. Rickman, M.W. Rowe, T.W. Boutton, J. Russ, J. and N. Guidon. 2002a. Accelerator Mass Spectrometry radiocarbon ages for an oxalate accretion and rock paintings at Toca do Serrote da Bastiana, Brazil. In *Archaeological Chemistry: Materials, Methods, and Meaning*, edited by K. Jakes, 22-35. Washington, D.C.: American Chemical Society.

Steelman, K.L., and M.W. Rowe. 2002. Potential for virtually nondestructive radiocarbon and stable carbon isotopic analyses on perishable archaeological artifacts. In *Archaeological Chemistry: Materials, Methods, and Meanings*, edited by K.A. Jakes, Chapter 3, 8-21. American Chemical Society Symposium Series 831. New York/Oxford: Oxford University Press.

2004. Non-destructive plasma-chemical extraction of carbon from organic artefacts. In *Radiocarbon and Archaeology* edited by T. Higham, C. Ramsey, and C. Owen, 35-42. Oxford, England: Oxbow Books.

2005. Dating pictographs: Independent dates and their implications for rock art. In *Making Marks*, edited by J.K.K. Huang and E.V. Culley, Chapter 2, 17-26. Tucson: American Rock Art Research Association.

2012. Radiocarbon dating of rock paintings: Incorporating pictographs into the archaeological record. In *A Companion to Rock Art*, edited by J.

McDonald and P. Veth, Chapter 32, 565-82. Oxford, United Kingdom: Blackwell Publishing Ltd.

Steelman, K.L., M.W. Rowe, R.F. Boszhardt, and J.R. Southon.

2001. Radiocarbon age determination of a rock painting at Arnold/Tainter Cave, Wisconsin. *Midcontinental Journal of Archaeology* 26: 121-32.

Steelman, K.L., M.W. Rowe, T.W. Boutton, J.R. Southon, C.L. Merrell, and R.D. Hill. 2002b. Stable isotope and radiocarbon analyses of a black deposit associated with pictographs at Little Lost River Cave, Idaho. *Journal of Archaeological Science* 29: 1189-98.

Steelman, K.L., M.W. Rowe, and T. Guilderson. 2005b. Non-destructive radiocarbon dating. In *Archaeology Between the Borders*, edited by M. Thompson, J. Jurgena, and L. Jackson, pp. 47-50. El Paso: El Paso Museum of Archaeology.

Steelman, K.L., M.W. Rowe, V.N. Shirokov, and J.R. Southon.

2002c. Radiocarbon dates for pictographs in Ignatievskaya Cave, Russia: Holocene age for supposed Pleistocene fauna. *Antiquity* 76: 341-8.

Steelman, K.L., M.W. Rowe, S.A. Turpin, T. Guilderson, and L. Nightengale. 2004b. Nondestructive radiocarbon dating: Naturally mummified infant bundle from SW Texas. *American Antiquity* 69: 741-50.

Steelman, K.L., E. Waltman, and M.W. Rowe. 2000. Feasibility of radiocarbon dating of pictographs from Piaui, Brazil. *SBPN Scientific Journal* 4: 90-7.

Taylor, R.E.

1987. Radiocarbon Dating: An Archaeological Perspective. New York, NY: Academic Press.

Terry, M., K.L. Steelman, T. Guilderson, P. Dering and M.W. Rowe.

2006. Lower Pecos and Coahuila peyote: new radiocarbon dates. *Journal of Archaeological Science*, 33: 1017-21.

Wacker, L., S.M. Fahrni, I. Hajdas, M. Molnar, H.-A. Synal, S. Szidat and Y.L. Zhang. 2013. A versatile gas interface for routine radiocarbon analysis with a gas ion source. *Nuclear Instruments and Methods in Physics Research B*, 294: 315-9.

Wood, Rachel.

2015. From revolution to convention: the past, present and future of radiocarbon dating. *Journal of Archaeological Science* 56: 61-72.

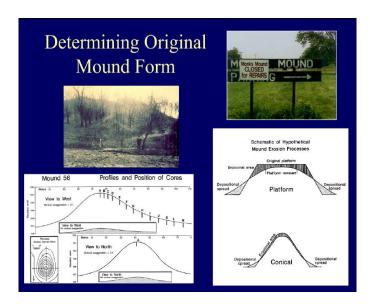
Rinita A. Dalan Minnesota State University Moorhead

#### **Abstract**

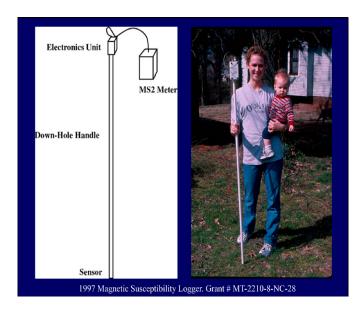
A 1997 NCPTT grant to develop a prototype downhole magnetic susceptibility instrument arose out of frustration with existing technology and a desire to expand archeological field studies of magnetic susceptibility. This instrument allowed highresolution vertical investigations of susceptibility within a small diameter (ca. 2.5 cm) hole made with a push-tube corer. An NSF grant supported improvement of the prototype via robust laboratory and field testing, resulting in a final engineered product (the MS2H) in partnership with Bartington Instruments, and also established an archaeological soil magnetic laboratory to improve research and training. A second NSF grant extended equipment and software, allowing increased integration of field and laboratory geophysical studies. Two additional NCPTT grants addressed the last crucial step in the advancement of down-hole susceptibility technology, namely application within archaeological practice. The first advanced the instrument's use in the detection of buried archaeological sites, and the second focused on the identification of unmarked graves. Due to its broad applicability, use of magnetic susceptibility technology has steadily grown. Integrating down-hole and laboratory techniques with surface geophysical surveys has produced a more mature magnetic susceptibility method that is much more widely employed than it was in 1997



The initial NCPTT grant application stemmed from frustration with existing technology for recording magnetic susceptibility in archaeological field applications (in particular the lack of instrumentation that allowed documentation of how susceptibility varies with depth) and the procedure that I was using to measure vertical variation in soil magnetic susceptibility. At that time, unless I was lucky enough to have an exposure or an open excavation, I was using an Oakfield push-tube corer to sample subsurface soils, cutting up the cores into segments and dropping those segments into a labeled muffin tin, bagging the samples, transporting them back to the lab, packing them in non-magnetic containers, and then measuring susceptibility on a Bartington MS2B lab sensor.

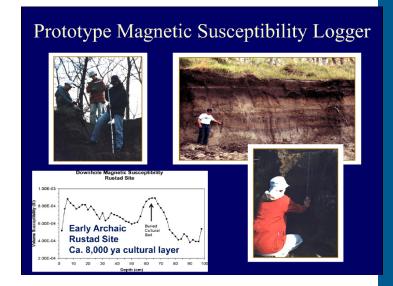


The reason that I was willing to invest in such a laborious process was that I knew that an understanding of how susceptibility changed with depth had immense potential for archaeological application. For example, I was working on a project at the Cahokia Mounds State Historic Site that involved using the relative thicknesses of magnetically enhanced topsoils, together with an understanding of hillslope processes, to determine the original forms of mounds at the site. In this and other projects, I was hampered by an inability to collect this information quickly and efficiently.



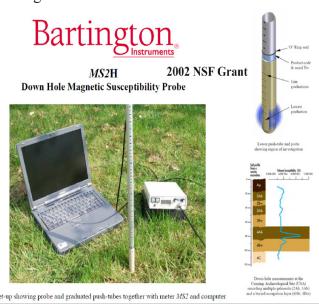
The NCPTT grant allowed me to rehouse

a Bartington Instruments MS2F sensor, designed primarily for investigating flat surfaces and sections, so that it could be lowered down a small diameter (ca. 2.5 cm) core hole to measure, in the field. susceptibility variation with depth. The sensor was housed in in a magnetically clean, watertight, electrically and mechanically secure mount and shaft constructed of PVC stock and pipe. Three cycles of instrument modification were completed before a prototype suitable for calibration was achieved. Readings were accomplished by connecting the sensor to a Bartington MS2 susceptibility meter, thus providing a compatible product for anyone who had access to a Bartington meter. The sensor was calibrated, to allow readings to be converted to SI volume susceptibilities, using a specially-constructed doughnut-shaped tank filled with material of a known susceptibility. In addition, the calibration tank was used to gather data on edge effects and measurement volume. As a mother with young children, a babysitter provided the needed time to work on this project.



Field trials were used to evaluate how the instrument would function in an archaeological field situation replete with distinct layering. Originally, a field trial at the Cahokia Mounds State Historic Site was planned but, due to a move to Minnesota State University Moorhead (MSUM), field trials ended up being completed at the Early Archaic Rustad site (32RI775) in southeastern North Dakota. The site had been excavated by Michael Michlovic, chair of

my new department, and remnants existed around the rim of a soil quarry. An Oakfield corer was used to make a hole approximately 15 cm behind the face of the pit. Once down-hole tests were completed, the face was cleaned back to within 5 cm of the cored hole. Down-hole results were compared to the stratigraphic section and to susceptibilities measured on samples taken from the cleaned face. The prototype instrument clearly identified the buried Early Archaic layer, more than 10 times faster than sampling from an exposure or through coring and measuring susceptibility in the laboratory, thus greatly expanding field capabilities for investigating variations in magnetic susceptibility across archaeological terrains.



The next step in developing down-hole magnetic susceptibility technology appropriate for archaeological and other near-surface applications was a 2002 NSF major research instrumentation development grant to produce a commercially available instrument. This involved a partnership with Bartington Instruments who engineered and produced the sensor. I was the unbiased tester and applications consultant, providing feedback from field and laboratory trials directed toward evaluating the performance of second-generation prototype instruments. The final engineered

product was the Bartington MS2H Down Hole Magnetic Susceptibility Probe. This sensor was fully integrated into the Bartington MS2 susceptibility system.



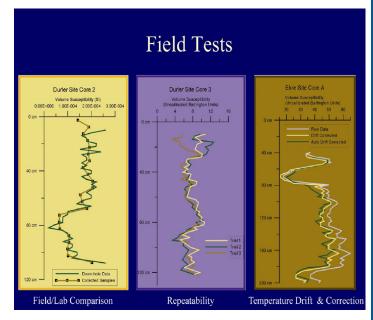
The route to a final engineered product was not always straightforward. Bartington initially floated some alternative designs, including this springy tubular housing being demonstrated by former MSUM student Amanda Butler. This particular prototype, which I received just prior to a scheduled demonstration at the 2002 Southeastern Archaeology Conference meetings in Biloxi, Mississippi, was certainly a challenge to present with all seriousness.



Eventually we settled on something more like the prototype constructed as part of the NCPTT grant, and various versions of this were produced and tested. A number of improvements were sought in the development of these second-generation sensors, including a more temperature-stable sensor, increased penetration depth, a segmented staff, and a data-logging system.



Undergraduate students from MSUM and I tested these prototypes against each other and the original instrument developed under the NCPTT grant. These tests were used to provide advice on further improvements. Performance was evaluated into terms of number of variables including repeatability (noise), sensitivity, stability, and resolution. In addition, the speed and ease of field operation, as well as flexibility of application, were considered.



Field tests provided the real-world experiences so necessary in final product design. Shown here are fields tests assessing repeatability, instrument drift, and accuracy compared to laboratory measurements using collected samples. Field tests were conducted at a number of sites in the southern Lake Agassiz region of Minnesota and North Dakota, providing varied conditions for assessing how well the logger was able to resolve stratigraphic sequences, soil horizons, and buried soils. These sites spanned a variety of soil types and ages.





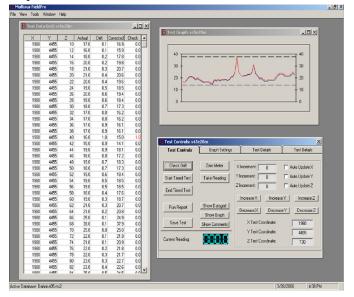
### **Laboratory Tests**

- 1) Drift/Temperature Response
- 2) Noise
- 3) Edge Effects
- 4) Sensitivity
- 5) Resolution

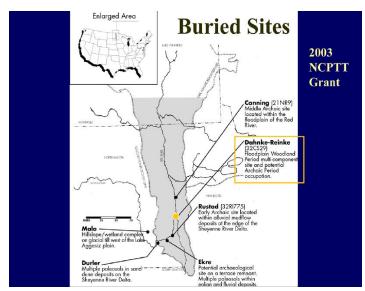


We also performed a host of controlled experiments in the laboratory using mixtures of

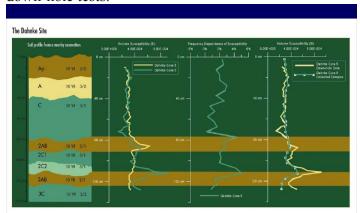
materials representing the range of archaeological possibilities. We looked at temperature drift (magnitude and timing), noise, edge effects, sensor sensitivity, layer resolution, etc. The data from the laboratory tests was also fed back to Bartington Instruments along with recommendations for improvement.



We even developed a prototype software package (MultisusFieldPro, which I still use today!). This software, developed for a Panasonic toughbook, allowed us to see data real-time in the field. Not part of the original NSF development grant, this effort was funded by Bartington Instruments, who supported an MSUM computer sciences student, Kim Humble, in developing the software. This database software can be used to control any of the Bartington MS2 field sensors moving in either the X,Y, or Z directions, records information on all settings and other field notes, and exports files into excel format.



Once a commercial instrument was available, a second NCPTT grant was obtained to establish the instrument within archaeological practice, in this case finding and mapping buried archaeological sites by identifying magnetic changes that occur as part of soil development and cultural occupation. Testing initiated as part of the NSF development grant was expanded utilizing a number of archaeological and other sites within the Red River Valley region of Minnesota and North Dakota. Locations with paleosols, but no archaeological remains, served as controls. Existing soil physical, chemical, and chronologic data were supplemented with additional soil cores and analyses for comparison with the down-hole tests.



The Dahnke site is comprised of two Woodland period layers.

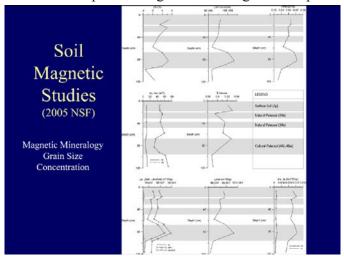
Soil derived from 1987 excavation block near the location of down-hole tests

Susceptibility peaks coincide with buried horizons

(2AB more prominent at Loc 3 and 3AB more prominent Loc 5)

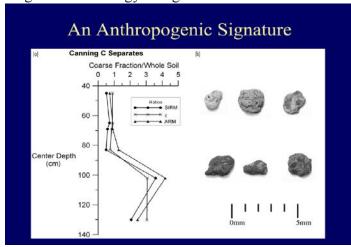
Downhole is more effective than collection and lab measurement

This figure shows data from one of the archaeological sites, the Dahnke site, a stratified site that contains at least two buried Woodland period layers. The soil profile used for comparison derives from nearby 1987 excavations and the graphs show results of downhole tests at two locations adjacent to this excavation unit. Susceptibility variations documented by the down-hole tests compare well with stratigraphic layers, and localized increases in susceptibility coincide with the buried horizons. Not only was the downhole more efficient than collecting samples for susceptibility measurements in the laboratory (with a measurement time of approximately 1 second, a hole could be logged in less than 1 minute), the small depth increments measured with the logger (in this case 2 cm) were more effective for identifying buried soils than collected samples homogenized over greater depths.

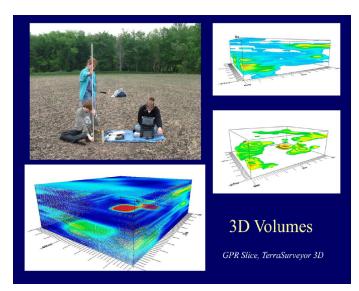


Another interesting find of the buried sites research was an apparent contrast between paleosols associated with human occupation and those that were not. Although both clearly correlated with relatively high susceptibilities, the magnetic susceptibilities of the buried archaeological soils were in some cases over twice the magnitude of the buried non-cultural soils. A second NSF grant allowed the purchase of additional soil magnetic instruments that were used to understand the enhanced magnetic susceptibility of these buried soils. Although differences in the magnitude of

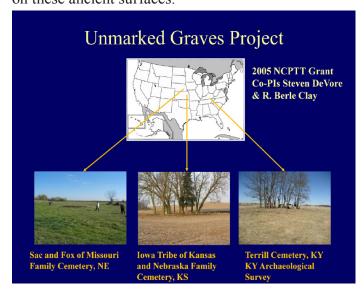
susceptibility enhancement suggested a possible avenue for determining if ancient land surfaces had been occupied by humans, efforts were directed toward exploring whether there might also be a distinctive signature for archaeological soils in magnetic mineralogy and grain size.



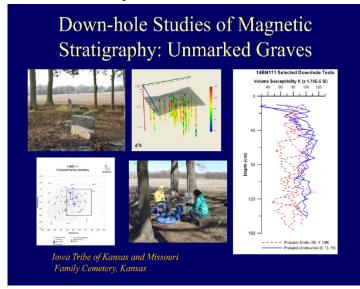
Work conducted as part of the NSF grant indicated a similar soil-forming process occurred in both the buried archaeological and non-archaeological soils, producing a finegrained magnetite and maghemite responsible for the enhanced susceptibility signal. The buried archaeological paleosols, however, showed a distinctive anthropogenic signature residing in the coarse fraction of these soils, potentially useful for separating buried soils from buried occupation layers. This signature related to an increase not only in superparamagnetic (SP) grains, but also a distinctive increase in grains at and above the SD (single-domain) grain size boundary. Nodules of soil, perhaps formed as a result of domestic fires, were visually and magnetically identified as the source of the distinctive remanence signal.



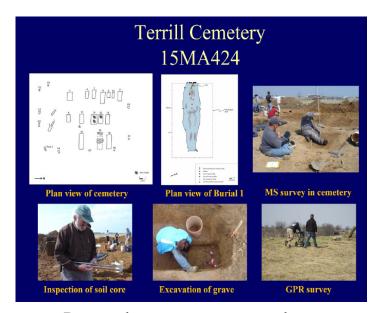
Because the MS2H was so fast and because it was so effective in providing information on buried soils and subsurface stratigraphy, we started using it to study entire landforms. We would complete a grid of down-hole tests to see how buried surfaces draped themselves across the landscape. To do this we moved into building 3D volumes of susceptibility with colleagues Dean Goodman (using GPR Slice) and David Wilbourn (using Terrasurveyor 3D). Constructing isosurfaces using these 3D volumes even allowed us to look for "hot spots" of occupation on these ancient surfaces.



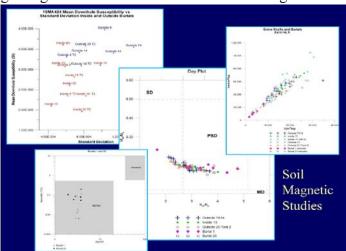
A final NCPTT grant, with Co-PIs Steven De Vore and Berle Clay, focused on another application for the down-hole sensor, confirming and improving surface geophysical results in the search for unmarked graves. Near-surface geophysical techniques have been primary tools in the detection of unmarked human internments, however they do not offer foolproof detection of all, or often even most, graves. Partners on this project were the Sac and Fox of Missouri and the Iowa Tribe of Kansas and Nebraska who allowed us to test this approach at two Native American family cemeteries in Nebraska and Kansas, and the Kentucky Archaeological Survey who allowed tests at an Anglo-American cemetery in Kentucky that was being excavated in advance of development.



Down-hole tests focused on grave shafts (not the burials), as located from surface geophysical anomalies and the presence of headstones and footstones. These were compared to down-hole tests in areas adjacent to the shaft, potentially representing undisturbed ground. A distinctive low-susceptibility signal of the shaft was observed at both the Native American family cemeteries. At select locations, samples were collected when making the hole for the down-hole sensor, and these samples were analyzed in the laboratory using a number of magnetic techniques.

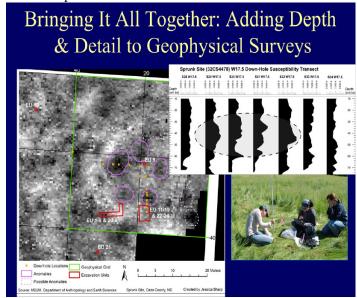


Because the graves were excavated at the Terrill Cemetery in Kentucky, the context of the down-hole tests (whether in a grave shaft or undisturbed ground) was secure. The pattern of a low-susceptibility grave shaft was also observed at this cemetery. At this cemetery, we were also able to investigate magnetic characteristics of the graves themselves, which indicated a pattern of magnetic enhancement useful for burial identification where grave goods and skeletal remains are lacking.



Soil magnetic studies indicated that the low susceptibility signature of the grave shafts was due to a lower concentration of magnetic minerals, in turn related to the lower density of soils refilling the shaft. Penetrometer studies, conducted at the Native American cemeteries as part of this grant, did not

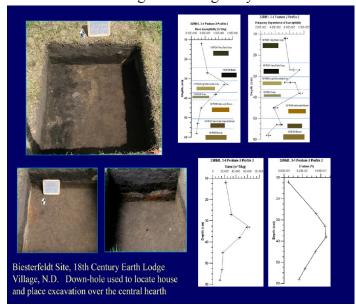
consistently identify the grave shafts, although this may be related to the insufficient depth sampled by the penetrometer. Though the magnetic signature of the grave shaft was not invariable, combining near-surface geophysical, down-hole magnetic susceptibility and soil magnetics provides for improved capabilities in the identification, evaluation, and thus preservation of unmarked human burials.



As part of our presentations we were asked to comment on how our NCPTT grants (and in this case I have also considered the related NSF grants) have influenced how we operate now. Certainly I would have given up susceptibility studies a long time ago if I was still collecting samples in muffin tins. Instead, down-hole susceptibility tests have become an integral part of the geophysics I do. I rarely use surface geophysics alone, instead I employ the down-hole instrument to gain more detailed information about anomalies documented by surface geophysical surveys. The example shown here is from an enclosure site in the northern Plains called the Sprunk site. Surface geophysical surveys revealed a possible circular arrangement of house basins within this enclosure. A transect of down-hole tests across one of these possible house basins was used to determine the depth, thickness, and lateral extent of this feature

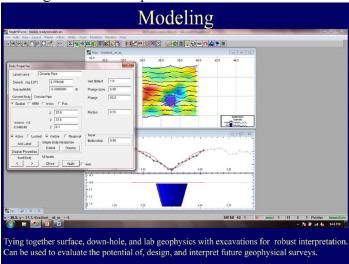


I also combine field studies of magnetic susceptibility with excavations whenever possible. Down-hole tests are used to precisely position excavation units, or to measure the depth of a feature being excavated or depth to sterile soils. I also use the Bartington MS2K sensor for both plan and profile maps of susceptibility. These maps often indicate the locations of features that are invisible, or not yet visible (will be defined in deeper levels) to excavators. Susceptibility maps within excavations allow me to directly connect geophysical properties to what we are seeing archaeologically.



For example, at the Biesterfeldt site, a proto-historic earth lodge village in North Dakota,

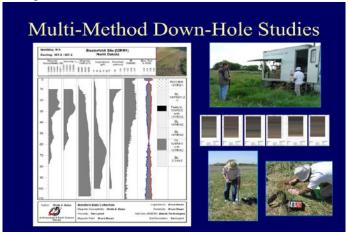
down-hole tests were used to confirm lodges in the plowed portion of the site where surface geophysical surveys only suggested possible locations. They were also used to place small excavations precisely over the central hearth of one of these lodges, where samples were collected for soil magnetic studies to investigate formation processes.



Gathering geophysical information on archaeological features also allows model building, using the depths and dimensions of features from excavations together with their geophysical properties. These models can be compared to surface geophysical surveys to see if they match, and, if not, can be used to explore why. They are also useful for evaluating the potential of and for designing and interpreting future geophysical surveys. Shown in this figure is a magnetic model of a post pit at Poverty Point constructed using excavation and magnetic susceptibility data and ModelVision Pro software.



Over all these years, the MS2H has been an amazingly useful and reliable instrument with wide applicability. What has required creativity and perseverance has been to make and hold open the hole to introduce the sensor. Coring has not been an insurmountable challenge, but it has required creativity and perseverance. Shown here are some of the options used to make these small diameter holes.



Of course once a hole has been made, it makes sense to use it to gather as much information as possible, including information on other geophysical properties. As shown in these tests at the Biesterfeldt site, my colleagues Bruce Bevan and Dan Lynch have experimented with various other electrical and magnetic properties in downhole applications, and Dakota Technologies, a Fargo company, tested their direct push color system at the site. Just like multiple-method surface geophysical surveys, combining several down-hole methods can

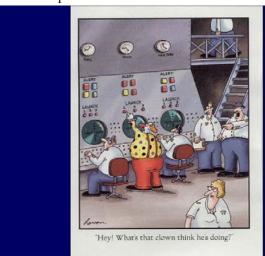
be very helpful in mapping subsurface remains.



In thinking about the disciplinary impact of this grant, I would refer to my 2008 review of archaeological magnetic susceptibility studies in the journal Archaeological Prospection. As I prepared that review I could see how field susceptibility studies in North American archaeology had matured since the 1990s, and since 2008 they have continued to develop. I think the annual National Park Service's remote sensing training course, in promoting the use of susceptibility instruments including the down-hole sensor, certainly has a lot to do with this. I must also credit the creative ways my colleagues have applied susceptibility studies in their work. Though field use of susceptibility instruments in archaeology is now well-established, there is still room for growth in soil magnetic applications to understand formation processes.



In preparing this paper, I had a lot of fun looking back at 17 years of pictures. I loved seeing pictures of undergraduate students at MSUM who worked with me on these grants; many, who now have graduate degrees, are using susceptibility studies in their own research, and are even teaching their students these methods. Certainly their participation in the instrument development process had an impact on their research and teaching.



In closing, I would like to extend my most sincere thanks to the National Center for Preservation Technology and Training for taking a chance that I could build an instrument useful for archaeological application. There are not many programs that support us in our dreams of a better way of doing our research. Thank you for this and for your great program.

### Acknowledgements

- NCPTT
- NSF
- Geoff Bartington & Bartington Instruments
- Steven DeVore, Mark Lynott and all my NPS Training Course Colleagues
- Institute for Rock Magnetism
- Students
- Collaborators

I would like to acknowledge the NCPTT and NSF for the support provided through grants described in this paper. I would like to thank the late Geoff Bartington and Bartington Instruments for their work in producing the MS2H sensor and in supporting development of archaeological studies of magnetic susceptibility. Many thanks are also due my colleagues at the Institute for Rock Magnetism for the technical support that they have continued to provide over the years. I am also indebted to Steven De Vore, the late Mark Lynott and the late John Weymouth, and all my NPS training course colleagues who have provided a forum for growing susceptibility studies and introducing the profession to the possibilities of this technology. Finally, thanks to all the students and collaborators, too numerous to list individually, who have worked with me on projects involving susceptibility studies.

### Grants

### **NCPTT**

- 2005 Identification of Unmarked Graves. Co-PIs Steven L. DeVore and R. Berle Clay. Grant Number: MAC/NCPTT Memorandum of Agreement; MAC/MSUM Cooperative Agreement HG115050086 Sac and Fox of Missouri, Iowa Tribe of Kansas and Nebraska, and Kentucky Archaeological Survey, Collaborators
- 2003 Development of a Technique for Buried Site Detection using a Down-hole Soil Magnetic Instrument. Grant #: MT-2210-03-NC-10
- 1997 Magnetic Susceptibility Logger. Grant # MT-2210-8-NC-28

### **NSF**

- 2005 Acquisition of Magnetic and Soil Magnetic Equipment for Ground-Testing and Interpreting Geophysical Surveys. Award # 0519749
- 2002 Development of a Down-hole Magnetic Susceptibility Logger and Soil Magnetic Laboratory for Archaeological and Soils Investigations. Award # 0215723

#### References:

Michlovic, Michael , George R. Holley, and Rinita A. Dalan.

Investigations at the Sprunk Site. North Dakota Archaeological Association. In Press.

Dalan, Rinita A., Bruce Bevan, Dean Goodman, Dan Lynch, Steven De Vore, Steve Adamek, Travis Martin, George Holley, and Michael Michlovic. 2011. The Measurement and Analysis of Depth in Archaeological Geophysics: Tests at the Biesterfeldt Site U.S.A. *Archaeological Prospection* 18: 245-25.

Dalan, Rinita A., Steven De Vore, and R. Berle Clay. 2010. Geophysical Identification of Unmarked Historic Graves. *Geoarchaeolgy* 25(5): 572-601.

Dalan, Rinita A.,

2009. Probing the Surface: Adding Depth and Detail to Surface Geophysical Surveys using Down-Hole Susceptibility Measurements. In Mémoire Du Sol, Espace Des Hommes, Archaeo Sciences: Revue d'archéométrie Supplément au n°33, 283-5. Presses Universitaires De Rennes.

Dalan, Rinita A.

2008. A Review of The Role of Magnetic Susceptibility in Archaeogeophysical studies in the United States: Recent Developments and Prospects. Archaeological Prospection 15: 1-31.

Dalan, Rinita A. and Dean Goodman. 2007. Imaging Paleo-Landscapes with Downhole Susceptibility. In Digital Discovery: Exploring New Frontiers in Human Heritage, edited by Jeffrey T. Clark and Emily M. Hagemeister, 279-85. Archaeolingua, Budapest.

Dalan, Rinita A. and Dean Goodman. 2007. Imaging Buried Landforms Using Down-Hole Susceptibility Data and 3D GPR Visualization Software. Archaeological Prospection 14: 273-80.

#### Dalan, Rinita A.

2006. A Geophysical Approach to Buried Site Detection Using Down-Hole Susceptibility and Soil Magnetic Techniques. Archaeological Prospection 13: 182-206.

#### Dalan, Rinita A.

2006. Magnetic Susceptibility. In Remote Sensing in Archaeology: An Explicitly North American Perspective, edited by Jay K. Johnson, 161-203. Tuscaloosa: University of Alabama Press.

#### Dalan, Rinita A.

2001. A Magnetic Susceptibility Logger for Archaeological Application. Geoarchaeology 16: 263-73.

Dalan, Rinita A. and Subir K. Banerjee. 1998. Solving Archaeological Problems Using Techniques of Soil Magnetism. Geoarchaeology 13(1): 3-36.

#### Dalan, Rinita A.

1997. The Construction of Mississippian Cahokia. In Cahokia: Domination and Ideology in the Mississippian World, ed. by T.R. Pauketat and T.E. Emerson, 89-102. Lincoln: University of Nebraska Press.

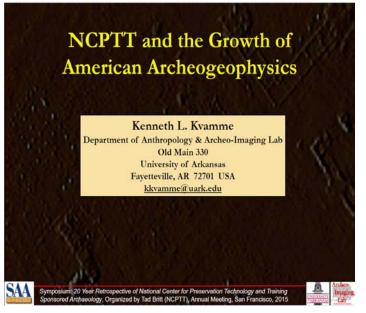
Dalan, Rinita A. and Subir K. Banerjee. 1996. Soil Magnetism, an Approach for Examining Archaeological Landscapes. Geophysical Research Letters 23(2): 185-8.

Dalan, Rinita A., Harold W. Watters, and Subir K. Banerjee.

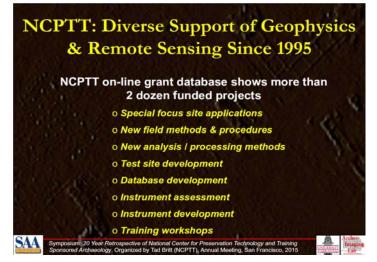
1996. The Application of Soil Magnetic Techniques for Determining the Original Form of Prehistoric Earthworks. Proceedings of the Sixty-Sixth Annual-Meeting of the Society of Exploration Geophysicists Vol. I, pp. 790-3.

## NCPTT and the Growth of American Archaeogeophysics

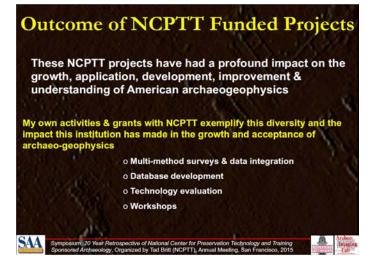
Kenneth L. Kvamme
Department of Anthropology & Archeo-Imaging Lab
University of Arkansas



I was very pleased and honored to be invited by Tad Britt of the National Center for Preservation Technology and Training (NCPTT) to participate in this symposium, in fact, to be one of a very few presenters to represent what the NCPTT has accomplished in promoting archeogeophysics in American archaeology.



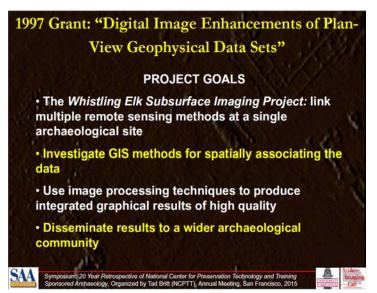
NCPTT offers an excellent website that is quite transparent about what it does. It includes a database covering all previous grants, and even a brief perusal indicates that NCPTT has been extremely supportive of the development and application of archaeological geophysics and remote sensing from its inception two decades ago. I counted more than two dozen grants in the diverse areas listed here that range from instrument development and assessment to new field and analysis methods, simple site surveys and applications, to the development of databases. Of equal significance is the support of training workshops that have promoted geophysical practices.



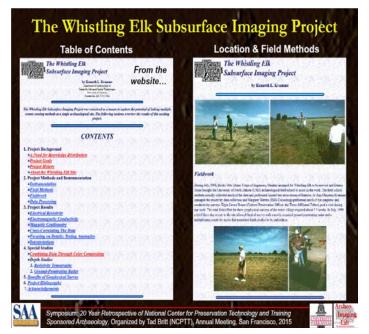
As a consequence, it is clear that the NCPTT has been a chief promoter of archeogeophysics and remote sensing, and much of its current success and acceptance may be attributable to NCPTT. It would be difficult for me to review all these past grants and projects and to do them sufficient justice. Rather, I will focus on my own grants and involvements with NCPTT projects as a means to illustrate some of the diversity of NCPTT support. I have had three grants with NCPTT for geophysical projects including one that explored multiple-method surveys and the integration of their data, another for the creation of a database of geophysical results, and a third for

#### NCPTT and the Growth of American Archaeogeophysics

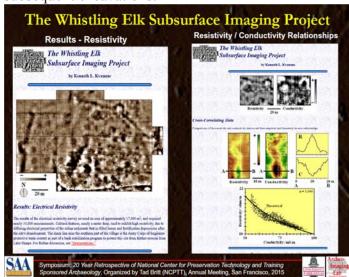
the evaluation of various instruments and findings in magnetic susceptibility surveys. I have also participated in several NCPTT sponsored workshops.



My earliest grant from NCPTT was in 1997 in a project I called "Digital Image Enhancements of Plan-view Geophysical Data Sets," an unfortunate title because it does not well explain the scope of this project. At that time an important issue in archeogeophysics was in the application of multiple geophysical methods to the same survey area and doing something more with the combined data other than merely displaying side-by-side maps of results. This project attempted to address these issues. As can be seen here several geophysical methods were applied to the Whistling Elk site in central South Dakota, a fortified Early Coalescent village (ca 1300 CE) buried nearly a meter below the surface. GIS methods were chosen as a means to manage the data and also to associate or integrate them in a more useful way. GIS also permitted various image processing methods to be employed. Finally, dissemination included the creation of a major website as well as conference papers and publications.

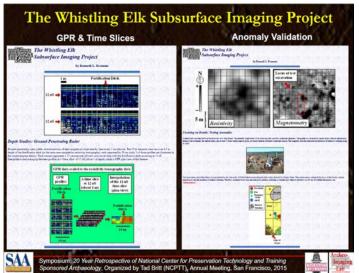


The next few slides portray some of the principal web pages for the project, including the table of contents. Assisting in the project was the University of North Dakota archaeological field school, directed by Dennis Toom. These students did much of the physical work and also performed subsequent excavations.



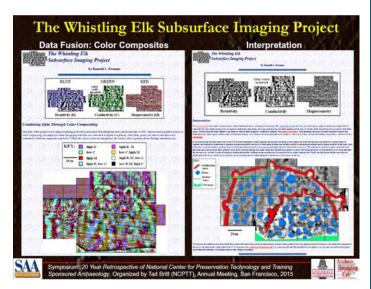
The geophysical results at the site were excellent. Here we see the electrical resistivity results from a Geoscan Research RM15 twin-probe array that portrays the outer village defensive ditch with 5 bastions, plus indications of numerous houses irregularly distributed within the village

(the Missouri River lies to the immediate south). An electromagnetic conductivity survey with the Geonics Ltd. EM-38 produced another highly correlated data set (conductivity is the theoretical inverse of resistivity) that permitted empirical correlation studies and assessments of the utility of each device. This study was one of my first comparisons that revealed that although the EM-38 is about 8 times faster than a twin-probe resistance meter, results tend not to be as clear or defining in moderately dry to very dry soil conditions.



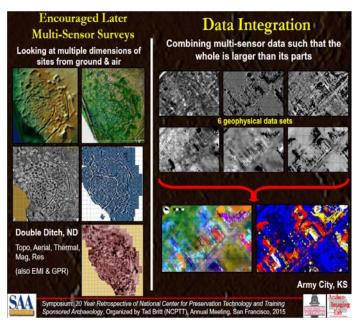
Limited ground-penetrating radar (GPR) was also employed in an attempt to clarify indicated features "stratigraphically" in GPR profiles or radargrams and also to explore them in relatively new time-slicing software that was then becoming available.

One of the principal anomalies in multiple data sets suggested a larger square "house" with southeast-facing linear entryway, probably the "ceremonial lodge" known from previous work at other sites and through ethnographic analogs. The geophysics indicated the central hearth, the loci of its four support posts, and the likelihood that the house was burned. A 2 x 6 m trench was excavated by Toom and his students that validated these inferences by exposing the hearth, one of the support post molds, and a perimeter that indicated burning.

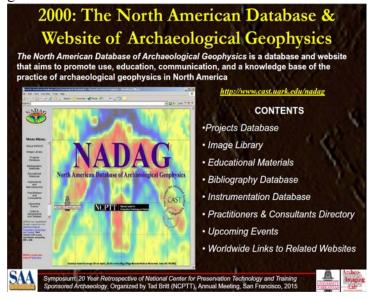


The data from the project were loaded into the Idrisi GIS that permitted image processing and enhancements (high- and low- pass filters, contrast improvements, statistical analyses that included correlational studies, differencing, and even principal components analyses). One form of data integration that proved particularly effective was simple color compositing, where red, green, and blue colors were assigned to three data sets which were then merged into a single color composite that permitted enhanced and rapid interpretations based upon understanding of the RGB color model. GIS also allowed digitization or vectorization of anomalies to graphically illustrate interpretations of the village through the geophysical results. Close inspection of the data offered the interpretation of a second, tighter village within the larger one with its own fortification ditch and a more densely packed collection of anomalies representing houses.

#### NCPTT and the Growth of American Archeogeophysics

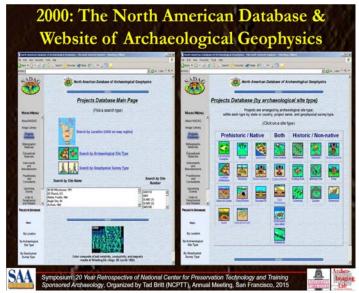


In the decade subsequent to the Whistling Elk Project the topic of GIS management and multisensor data integration became a popular one in the field of archaeo-geophysics worldwide. My later projects at the Double Ditch site in North Dakota (1490-1770 CE), which I carried out from 2001-2004, and at Army City in Kansas (1917-1922 CE), undertaken in 2004-2006, were heavily involved with GIS and data integration methods following very much from the path initially started by my grant from NCPTT.

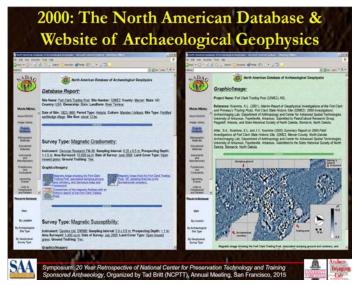


In 2000, believing in the promise and ultimate growth of archeo-geophysics, I won a multi-

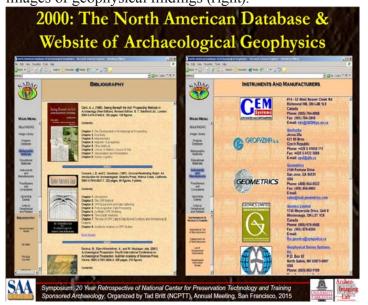
year grant from NCPTT to establish the "North American Database of Archaeological Geophysics" or NADAG (http://www.cast.uark.edu/nadag). The project was ambitious for its time with goals of establishing a database of basic results from past projects, geophysical imagery from those projects, databases including bibliographic citations, instrumentation descriptions, directories pointing to practitioners, lists of upcoming events, educational materials and more.



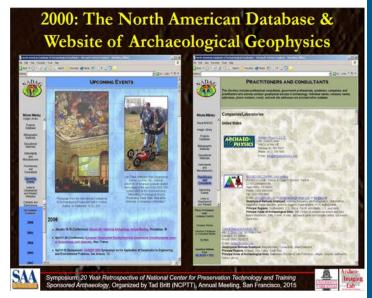
At that time, most of the coding was in basic HTML. A front page in the geophysical database section permitted searching by location, site type, by kind of geophysical survey, by site name, or number. The right page illustrates icons representing categories of archaeological site types in the site search menu.



The core database focused on archeogeophysical reports (left). What I thought would grab the interest of most archaeologists was an "Image Database" showing through graphical portrayals images of geophysical findings (right).



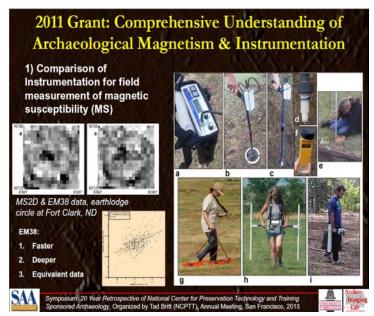
In additional to a searchable database of citations from publications and gray (CRM) literature, great detail was given to published books with complete tables of contents (left). Pages were also devoted to instrument manufacturers with basic contact information (right).



Upcoming events were listed by year with links to associated web pages and in another database, practitioners and consultants in archeogeophysics were listed.

Unfortunately, this project faced a number of unforeseen difficulties. A major one was a lack of response among potential contributors who pointed out a frequent inability to submit materials owing to lack of time and to the needs of clients who did not want results freely disseminated. Obtainable gray literature reports frequently contained poor quality imagery in photocopy formats, and published results could not easily be included in the database owing to copyright restrictions. The largest limitation, and an insurmountable one, was a cutting of funding by NCPTT due to general federal budget cuts at the start of the Iraq War, a circumstance which inhibited further development, although interested students and myself continued to maintain the database.

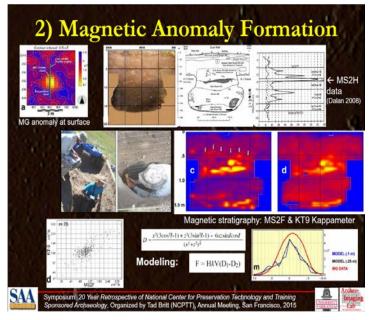
### NCPTT and the Growth of American Archeogeophysics



I pursued my final grant with NCPTT in 2011 with my Ph.D. student, Adam Wiewel at the University of Arkansas. In addition to confronting four focused research questions in archaeological magnetic prospecting, data gathered by this project was of great importance to his dissertation project. The project title, "Comprehensive Understanding of Archaeological Magnetism and Instrumentation," was perhaps pretentious in that it did not yield a comprehensive understanding of archaeological magnetism and all of its instrumentation, but it did make large in-roads to understanding in four major domains

The first research question focused on the comparison of instrumentation for the field measurement of magnetic susceptibility (MS) from the surface. It compared (1) a twin coil device, the Geonics Ltd. EM-38, with the single coil Bartington MS-2D, for gathering data in area surveys through a grassy surface at the Fort Clark State Historic Site in North Dakota (1822-1861 CE). It also compared (2) the Exploranium KT-9 Kappa meter against the Bartington MS-2F for acquiring MS data from excavated wall profiles at the Double Ditch State Historic Site in North Dakota (1490-1770 CE). Several sites, features, or wall profiles were surveyed

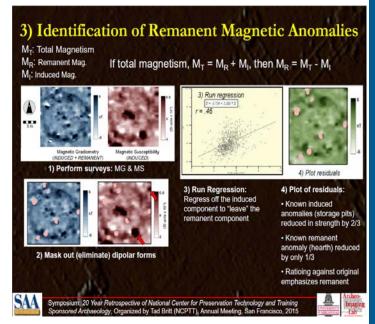
with each instrument and in the first study the data were also compared against magnetic gradiometry findings. The area surveys of the first study showed roughly equivalent data with similar anomalies indicated (although their correlations were only moderate with r about 0.5), but the EM-38 was about 10 times faster than the Bartington MS-2D and it permitted greater depth penetration to about 50 cm, compared to the latter's 10 cm. The comparisons of wall profile data in the second study (next page) showed both instruments to perform very similarly (with high correlations around r=0.8).



The second research question focused on anomaly formation and was the most complex. A magnetic gradiometry survey conducted at the Double Ditch State Historic Site from 2001-2004 yielded thousands of anomalies, many of which were excavated from 2002-2005. With the aid of the State Historical Society of North Dakota under the supervision of Fern Swenson, several of the excavations at known features (a storage pit shown here, a fortification ditch, and a house floor, all in cross-section) were reopened, and the walls were scraped clean. The stratigraphy of each wall was measured and mapped, and each wall was digitally photographed (these photographs were later composited). MS measurements were then acquired

every 5 cm by the Exploranium KT-9 Kappa meter and the Bartington MS-2F, except on the large profile that traversed the fortification ditch, which was measured every 10 cm. These data permitted mappings of the magnetic stratigraphy, with parallel results shown by both instruments (r=0.8).

This slide shows the "discovery anomaly" of a subterranean storage pit yielded by the magnetic gradiometry survey of 2001 with a Geoscan Research FM-36 (upper left), together with the photomosaic of the pit in cross-section and its interpreted stratigraphy. Down-hole MS recorded every 2 cm with a Bartington MS-2H through the center of this pit in 2005 by Rinita Dalan of Minnesota State University Moorhead (upper right). It shows basket loading of materials of varying MS as the pit was filled after its abandonment. MS data were recorded across the face of this profile every 5 cm yielding magnetic stratigraphy (middle row). The last indicates the nature of subsurface magnetism; the question then becomes, "how does this magnetism combine to form an anomaly recorded by a magnetic gradiometer at the surface?" Mathematical modeling was employed to address this question, and a moderately good fit was obtained between the modeled results and the actual magnetic gradiometry measurements over this profile. Lack of fit in this case is probably partially due to the presence of basket loads of fired clays dumped into the pit that likely contained materials with thermoremanent magnetism (they also illustrate very high MS seen as the bright spots in the middle of the magnetic profiles), which is not recorded by the MS instruments. The fit between other data and the models (not shown here) was closer because no thermoremanent materials were present.

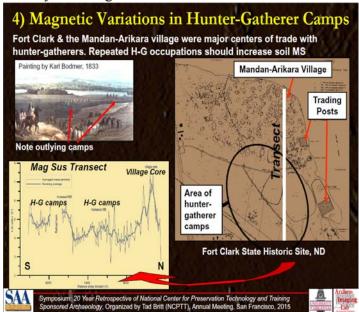


The third study examined the possibility of better isolating thermoremanent anomalies through several surveys at the Fort Clark State Historic Site (1822-1861 CE) and at Double Ditch (1490-1770 CE). Since a magnetic gradiometer (MG) records total magnetism, both from remanent and induced sources, while a MS meter records only the latter, one may "subtract" the MS data through a regression approach that isolates residuals to better define remanent anomalies that generally arise archaeologically from thermoremanence or intensive firing.

The approach illustrated here shows its promise and also its shortcomings. Anomalies known to arise from induced magnetism, the house perimeter and storage pits, were reduced in magnitude in the residual data by about 2/3 while hearth anomalies illustrating thermoremanence were reduced by only 1/3 (firing also increases MS or the induced component), making them much more visible. The shortcoming of the approach was the relatively low correlation (r<0.5) between the MG and MS data which reduced the power of the regressions. Part of the low correlation was due to the imprecise registration of the data. In other words, the exact measurement points of the MS and MG

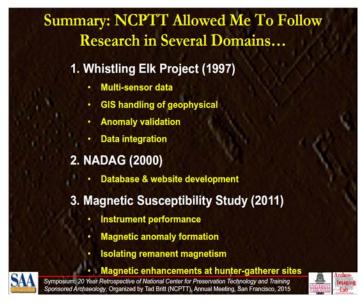
### NCPTT and the Growth of American Archeogeophysics

data were not perfectly coincident particularly in the vicinity of strong anomalies.

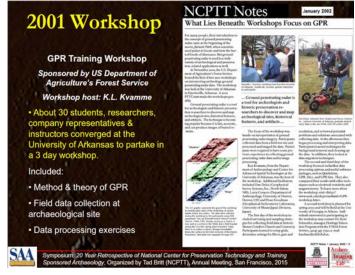


The final study explored magnetic variation in the vicinity of known hunter-gatherer camping areas reported in the historic literature in the vicinity of Fort Clark and Fort Primeau, two historic trading posts in the Fort Clark State Historic Site (1822-1861 CE). Geophysical surveys typically focus only on past cultures with built architecture that causes large and detectable disturbances to the ground. As hunter-gatherers were nomadic and without architecture, their sites are rarely explored geophysically. However, it is well-known that any human occupation through time tends to increase the magnetic susceptibility of the soil, and the grounds around the trading posts and large Mandan-Arikara village of horticulturalists were occupied annually by hunter-gatherers throughout the length of the their histories.

A series of transects across the park area were therefore run with MS instrumentation that showed unequivocally magnetic enhancement in the known areas of the hunter-gatherer camps.



My own funding by NCPTT has permitted growth of geophysical knowledge in a wide variety of domains in each project, and I think these projects well illustrate the kinds of diversity that NCPTT has supported in all of its grants in the field of archeogeophysics.



I also participated or hosted workshops in geophysical training that were funded or partially funded by NCPTT. The first, in 2001, was one sponsored by the U.S. Department of Agriculture Forest Service and funded largely by NCPTT (with Kent Schneider chief promoter). I was the host and organizer at the University of Arkansas where we outfitted a room for lectures, a lab for computer exercises, and actually collected data at a nearby

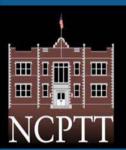
historic archaeological site that held the foundations of a church destroyed in the Civil War. Several leaders in archaeological GPR, including Dean Goodman, Larry Conyers, Margaret Watters, and Dan Delea, as well as instrument manufacturers such as Geophysical Survey Systems, Inc, participated in the instruction.



In 2012 I was delighted to participate in another NCPTT funded training workshop in archaeological geophysics that was organized by Margaret Watters and held at Washington's Headquarters and Longfellow House National Historic Site in Cambridge, Massachusetts. Margaret Watters led the instruction in all geophysical methods with myself, Bryan Haley, and Stephen Wilkes assisting, the latter in three-dimensional laser scanning with equipment provided by Feldman Land Surveyors of Boston.



To summarize, NCPTT has been instrumental to the growth of geophysical prospecting in American archaeology. Dozens of its grants have permitted development of new instruments and new field and analysis methods. I believe NCPTT has promoted "pure research" in archeogeophysics more than any other American institution. On top of that, new fieldwork has also been promoted, databases have been developed, and training workshops have been sponsored, all of which have contribute to the growth of geophysical applications in this country.



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